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REPORT _____

PHOSPHONITRILIC FLUOROELASTOMER COATED FABRICS FOR
COLLAPSIBLE FUEL STORAGE TANKS

Richard W. Sicka and George B. Mitchell
Firestone Central Research Laboratories
The Firestone Tire and Rubber Company
Akron, OH 44317

30 March 1979

Final Technical Report
Contract Number DAAG46-78-C-0006

Prepared for:

U. S. ARMY MATERIALS AND MECHANICS RESEARCH CENTER
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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This report describes the preparation of fuel resistant coated fabric materials that are serviceable at temperatures as low as -70°F. The coated fabric materials were made from a phosphonitrilic fluoroelastomer compound coated on nylon fabric. Efforts were directed toward attainment of calenderable compounds and test evaluations of the coated fabric. High filler loaded materials proved to be better processable materials. These | | | |

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showed good diffusion rates with test fluids (TT-S-735, Type II and Fuel S). Even better diffusion rates were seen with Arctic diesel fuel. The low temperature flexibility of the vulcanizates and coated fabric was promising for PNP-200 compounds. The seam adhesion strength and the fabric-to-rubber adhesion need to be improved before utilization of this material in tank constructions.

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SUMMARY

A critical need exists for fuel resistant materials which will function at temperatures of -57°C (-70°F) for application in self supporting 3,000 and 10,000 gallon collapsible fuel storage tanks.

This work is part of a continuing effort to develop materials which can form the elements of a refueling and storage system capable of service under Arctic weather conditions.

The initial phase of this work was to evaluate the PNF[®]-LT and PNF[®]-200 materials as candidates for coated fabric materials with which collapsible fuel tanks could be fabricated. Consideration of PNF[®]-200 was dictated in part by the need for low fuel diffusion rates. Fuel diffusion studies of the hose and tube compounds of PNF-LT used in the fuel hose contracts showed excessive rates of fuel diffusion for a coated fabric to function as a storage vessel material. Compounds of low filler content PNF-200 materials coated on nylon fabric showed promising values of low fuel diffusion rate and sufficient flexibility at low temperature (-70°F) to warrant consideration as a coated fabric candidate. Processing difficulties prevented their use as calenderable materials. Higher filler loaded materials proved to be better processable materials and showed good diffusion rates with test fluids (Type II, Fuel δ) and even better rates with Arctic diesel fuel.

Adhesion to fabric is an area requiring further study and development.

PREFACE

Investigations performed under Contract DAAG46-78-C-0006 from 1 February 1978 to 30 March 1979 are described in this final report. The primary objective of this contract was the development of phosphonitrilic fluoroelastomer vulcanizates and coated fabrics for use in the manufacture of self supporting 3,000 and 10,000 gallon collapsible fuel storage tanks with a -57°C (-70°F) capability.

This final report was prepared by the Central Research Laboratories of The Firestone Tire and Rubber Company. The work was sponsored and administered by the U.S. Army Materials and Mechanics Research Center, Watertown, Massachusetts. Dr. Robert E. Singler served as the Contracting Officer's Technical Representative.

In addition to Dr. Singler, we also wish to acknowledge Mr. Angus Wilson of U.S. Army Natick Research and Development Command and Messrs. Paul Touchet and Paul Gatzka of the U.S. Army Mobility Equipment Research and Development Command for their helpful discussions relevant to the objectives of this program.

Project management at Firestone was provided by Dr. D. P. Tate, Assistant Director of Research and Dr. A. E. Oberster, Research Associate. Many co-workers at the Firestone Central Research Laboratories assisted in the compounding and testing phases of this investigation and their support is gratefully acknowledged.

1.0 INTRODUCTION

The goal of this program was the development of PNF[®] phosphonitrilic fluoroelastomer compounds suitable for vulcanizates and coated fabrics for use in the manufacture of self supported 3,000 and 10,000 gallon collapsible fuel storage tanks. The military specifications MIL T-52573 and MIL T-43431 (ref. 1,2) served as guidelines except that a -57°C (-70°F) capability was desired. These tanks should satisfy the Army material requirements for refueling operations in the Arctic.

The U.S. Army Mobility Equipment Research and Development Center has sponsored earlier contract efforts for the development of PNF[®] fuel hoses (ref. 3, 4, 5). The first investigation (Contract DAAK02-73-C-0464) showed that fuel hoses could be fabricated from PNF-200 by a plied calendered sheet process. The hoses produced were not sufficiently flexible at -70°F (ref. 3). The second study (Contract DAAG53-75-C-0187) utilized a modified PNF[®], PNF[®]-LT which had better low temperature flexibility. This effort resulted in a hose with suitable flexibility when constructed of plied calendered sheets. The third program developed extrudable compounds for producing collapsible and suction type fuel hoses with hand wrapped covers. Large lengths of collapsible and suction hose were manufactured on Contract DAAK70-76-C-0239 and are under field test and evaluation in the Arctic.

The U.S. Army Materials and Mechanics Research Center recognized the need for collapsible fuel storage tanks capable of service at low temperatures and the potential offered by phosphonitrilic fluoroelastomers, as fuel resistant, extreme low temperature flexible coated fabric materials. The result is their support of this contract to develop PNF coated fabric materials.

PNF coated fabrics were prepared and examined by A. Wilson of Natick Laboratories for fire resistance comparison to MUST shelter coated fabric (ref. 6). MUST is an acronym for "Medical Unit Self Contained, Transportable." Although the current MUST shelter fabric uses commercial fire resistant rubbers, polychloroprene-top coated with chlorinated polyethylene, the Army experienced field incidents exposing the shelters to fire. A. Wilson cement coated PNF phosphonitrilic fluoroelastomer onto polyester fabric to prepare MUST-equivalent (14.8 oz/yd²) material. The phosphazene coated fabrics had a higher resistance to burning than currently used materials. The excellent low temperature properties of the PNF coated fabrics were also exhibited. Adhesion properties to fabric were found to need further development.

Collapsible fuel storage tanks have a long history of development (ref. 7, 8, 9, 10, 11, 12, 13) and include storage reservoirs of 5,000 barrel capacity. Early programs on design and development of such collapsible storage reservoirs have established design criteria that include water and environmental resistance of the material in addition to resistance to the aromatic content of the fuel. Further, the wide range of temperature requirements for service were expanded to include service, especially deployment in temperatures down to -70°F. Current materials have limited capability to -25°F.

Testing of POL tanks at the U.S. Army Tropic Test Center, Fort Clayton, Canal Zone, have identified (ref. 7) design and material problems associated with storage of fuels. Diesel fuel was more destructive to tank material than storage of JP-4. Design/material problems include rapid deterioration of the exterior coating allowing solar radiation and rain to attack the fabric and fuel. The seams appear to be a source of fuel leakage.

Fuel acidity increases in pH from 7.2 to 3.8 was suspected to be the major cause of degradation and was attributable to ultraviolet radiation and moisture.

Table 1 presents the desired properties of coating compounds and Table 2 presents the properties required for a collapsible fuel tank coated fabric. Low temperature flexibility, elongation, and fuel contamination values are lowered while the retained tensile properties have been increased over the MIL T. 52573C specification.

Table 3 gives a summary of the coated fabric properties and vulcanizate properties for two ranges of filler loading in PNF-200 formulations.

Generally, the fuel diffusion values are seen to be close to the desired values. Very low (< 0.01) values are observed in coated fabric which has used a heavy surface treatment. Fuel diffusion through PNF-200 vulcanizates are about 0.09 fl oz/sq ft 24 hrs for high filler levels and about 0.18 fl oz/sq ft 24 hrs for low filler level vulcanizates. The high filler vulcanizates show good retention of tensile properties on fuel aging at 160°F for 14 days. The vulcanizates also show good values for existent gum.

2.0 INVESTIGATION

2.1 Polymer

PNF-200 is a semi-inorganic fluoroelastomer commercialized by The Firestone Tire & Rubber Company. This phosphonitrilic fluoroelastomer has a service temperature range of -70°F to 350°F and possesses a unique combination of properties including solvent resistance and low temperature flexibility, high temperature stability and excellent mechanical properties.

PNF has excellent resistance to hydrocarbon fluids, lubricating oils and fuels. PNF is not suitable for use in oxygenated solvents such as ketones, ethers and alcohols. PNF-200 is useful for continuous service in most environments at 350°F. It can be applied for intermittent service at temperatures in the range of 400-500°F (ref. 14).

Typical PNF compounds have limiting oxygen indices in the range of 55-60. This makes PNF a candidate for use in elastomeric flame resistant coating and coated fabrics. A. Wilson has examined briefly the smoke and flame properties of PNF coated fabrics (ref. 6).

PNF-LT is a phosphonitrilic fluoroelastomer containing pendant fluoroalkoxy groups with a reduced level of fluorine content in the polymer. This illustrates one of the many variations possible with the polyphosphazene system. Such a modification lowers the Tg of the polymer at the expense of solvent resistance.

Table 4 shows the properties of a nylon fabric which has been used by Firestone in the preparation of other coated fabric tank material. This material meets or exceeds the properties desired in MIL T-52573C. Table 5 presents the physical property data on the various PNF polymers used in this program.

2.2 General Approach

The initial effort of this program concerned the selection of the type of PNF polymer: PNF-LT versus PNF-200. Initial compounding and testing was directed to evaluate the fuel diffusion and low temperature flexibility. Following this selection, additional GFM (government furnished material) was obtained and the questions of diffusion, processability and retention of properties on fluid aging received the most attention in further compounding.

Small mixes sufficient to prepare tensile ring specimens and to permit about 2 each 6" x 6" coated fabric samples to be built from a formulation were used to establish properties.

Large batches were Banbury mixed and calendering of stock and fabric was then attempted for the most promising mixes.

2.3 Experimental Details

2.3.1 Instruments

1. Laboratory Rubber Mills:

- a. 2" x 6", L. Albert and Son, Model A-6974
Capacity: ca. 100 g of PNF stock.
- b. 6" x 12", Farrel-Birmingham, Inc. Model 4-67.
Capacity: ca. 2 lbs. of PNF stock.
- c. 10" x 12" Farrel-Birmingham, Inc.
- d. 12" x 20" Farrel Co., Div. USM Corp., Inc.
Model A1500. Capacity: ca. 25 lbs. PNF compound.
- e. 3" x 7" Farrel-Birmingham Co. Model A1821.
Variable speed, individual roll drive.
Capacity: ca. 120 g PNF stock.

2. Calender, 4 roll inverted "L" 6" x 13". Variable speed. Capacity: ca. 10" wide sheet with roll guides in place.

3. Brabender Mixer, Model PL-V150. C. W. Brabender (CWB) Instruments, Inc. Capacity: ca. 120 g of PNF stock.

4. Banbury Mixer, Model "BR" A1548 Farrel Co., Div. of USM Corp, Inc. Capacity: ca. 2,200 g of PNF stock.

5. Laboratory Balances

- a. Mettler, Model PN2210, used for weighing pigments and polymer for small batches.
Capacity: 2,200 g, \pm .005 g.
- b. Toledo, Model 3710, used for weighing of pigments and polymer for large batches.

6. Instron, Model No. 1130, The Instron Corp. Used for stress-strain measurements. The instrument is interfaced with a Hewlett Packard Computer for computations of stress-strain data using micro-tensile rings. ASTM D3196.
7. Shore A Durometer. Shore Instrument and Mfg. Co., Inc.
8. Gehman Torsional Wire Apparatus. Wallace Test Equipment, Testing Machines, Inc. Amityville, NY.
9. Forced Air Oven, Blue M Electric Co., for heat aging polymer and post curing vulcanizates and coated fabric.
10. C. W. Brabender Electronic Plasti-corder Torque Rheometer, Model EPL-V751.

2.3.2 Mixing Techniques

In Brabender mixes, the polymer is added to the mixer and is consolidated at low speed (10 rpm) for about one minute. The reinforcing filler is then added while mixing at medium speed (40-60 rpm). Non-reinforcing fillers, MgO and stabilizer which are blended are then added. Complete addition of fillers occurs within about six minutes. The mixer is then run at about 110 rpm until an integrated torque of 20,000 meter-gm min. is attained or about 15 minutes total mix time. The mix is then dumped. Curing agent is then added to the masterbatch banded on a mill. Ambient temperature mills were generally used.

Better results of mixing were obtained with cooled mixing chambers.

Banbury mixing followed much the same procedure above: Cooling water on maximum flow.

0 minutes-load polymer, speed: slow (77 rpm)
 2 minutes-add fillers
 7 minutes-add stabilizer masterbatch
 15 minutes-dump mix

To obtain as low a mix temperature as possible additional cooling was supplied to the mixing chamber walls by dry ice. Temperatures as low as 150°F (65°C) were obtained.

The curative was added on a two roll mill with cooling water running in the cored rolls in order to maintain

a low temperature.

The several batches of a given formulation were mill blended together on the 10" x 20" Farrel mill.

2.3.3 Calendering Techniques

Initial test samples were prepared from small Brabender compounds by sheeting material on a vari-drive 3" x 7" mill where roll speed could be matched as on a calender. Sheets 0.020" thick and less were prepared and coated fabrics formed in 6" x 6" cavity molds suitably shimmed to appropriate thicknesses. These samples served as initial fuel diffusion and low temperature flexibility specimens.

A number of the compounds were difficult to handle by this procedure as the compounds did not have suitable green strength or tended to retract after release from the roll of the mill.

Late in the program a technique was developed which overcame this difficulty. This involved applying a sheet of .005" thick polyester film to the fast roll of the mill by means of double coated adhesive tape. The stock could then be worked on to the polyester and removed readily from the mill roll along with the polyester carrier sheet. Trimming the stock and polyester carrier to mold size provided convenient preparation of even difficultly processable materials.

Calendering on the 6" x 13" four-roll inverted "L" calender was accomplished to form 60 mil thick x 6" wide sheets which were roll-wrap cured between polyester film or metal shim stock for improved sheet finish and release. The most promising processable, yet impermeable to test fluid, stocks were calendered to sheets .025" thick x 10-1/4" wide in lengths to 25 feet. Initial attempts to calender the PNF stock into the 2 x 2 basket weave nylon fabric met with difficulty primarily due to the preferential adhesion of the stock to the calender rolls instead of the surface agent treated fabric.

This difficulty was surmounted by the use of carrier film material. The fabric was calendered into one sheet of .025" thick PNF stock and then the second surface of the fabric was calendered with stock on a second pass.

In each case carrier film material was interposed between the PNF stock and calender roll surface.

2.3.4 Physical Test Methods

Test specimens were prepared from sheeted samples by press curing 50 mil thick x 3" x 3" specimens. Coated fabric specimens were prepared in a 6" x 6" cavity mold. Calendered stock and coated fabric were wrap cured.

1. Stress-strain - ASTM D3196. Specimens were cut from 50 mil thick x 3" x 3" vulcanizate sheets.
2. Shore "A" hardness - ASTM D2240. Tests were made on plied vulcanizate.
3. Gehman low temperature measurements - ASTM D1053. Specimens 1.5" x 0.125" of vulcanizate were cut from sheets. An IBM 1130 computer was programmed for computation and print-out of Gehman data and graphs. Specimens 1.5" x 0.250" of coated fabric were cut from samples and similarly tested by ASTM D3388.
4. Fuel diffusion tests. Tests were conducted in accordance with para. 4.6.2.2.2 of specification MIL T-52573C with coated fabric samples and vulcanizates of promising PNF stocks.
5. Weather-O-Meter Atlas 18-WR used for accelerated aging of specimens with Cam No. 47, 18 hours of light and water in a 102 min. light and 18 min. water spray cycle followed by a dark period of 6 hours. Samples were aged for 500 hours total light. Black panel temperature $63^{\circ} \pm 5^{\circ}\text{C}$ ($50 \pm 5\%$ relative humidity) is obtained when illuminated.
6. Existent gum test performed in accordance with para. 4.2.1.1.1 and 2 of MIL T-52573C and ASTM D381.

2.3.5 Compounding

The initial phase of this program involved the evaluation of PNF-LT and PNF-200 materials as candidates for coated fabric materials with which collapsible fuel tanks could be fabricated. Fuel diffusion studies of the hose and tube compounds of PNF-LT used in the fuel hose contracts (ref. 3,4,5) showed excessive rates of fuel diffusion for a coated fabric to function as a storage vessel material. After consideration of the diffusion rates of TTS-735, Type II fuel through coated fabric samples prepared with PNF-LT, PNF-200 and blends thereof (see Tables 13, 14, 15), it was determined to confine the

remainder of the program to development of coated fabric material with PNF-200 based on its better diffusion characteristics. The low temperature flexibility of low filled PNF-200 compounds may be acceptable in the format of a coated fabric. Studies of low temperature torsional stiffness ratio provided evidence for this selection.

Table 6 presents the results of compounding each of the PNF-200 polymers with a typical low filler content vulcanizate and also a high filler content formulation. Low filler content materials generally show higher stress-strain properties, while the high filled materials exhibit better processability.

Various filler materials were examined at low filler loadings (15 phr). These include surface treated silica fillers, (Tullanox 500), surface treated anhydrous aluminum silicate, Burgess KE and clay fillers such as talc (Mistron Vapor). The compounds attempted are presented in the Tables 6 through Table 12. Table 3 summarizes the leading formulations of PNF-200. The fuel diffusion rates for similar compounds were determined and are summarized in Tables 13 through 15. The PNF-LT stocks show fuel diffusion rates which are markedly higher than similar compounds based on PNF-200. Blending in various amounts of PNF-200 did not improve the diffusion rate values significantly. PNF-LT was not considered further in the compounding program. The fuel diffusion rates of the early PNF-200 compounded materials are 0.07 fluid oz/sq ft/ 24 hours. This is close to the desired value but requires improvement. Fuel diffusion rates of coated fabric as low as <0.01 were obtained, (see Table 29).

Table 16 and 17 presents the results of G.C. analysis of the residual fuel in the test cups after a coated fabric diffusion rate determination. The PNF-200 stocks showed the lowest loss of material to be the iso-octane. The PNF-LT stocks had high losses of iso-octane from the fuel mixture.

The trend of weight loss of benzene > toluene > xylenes >> iso-octane was seen for PNF-200 stocks. For PNF-LT the trend was the same but the magnitude of iso-octane loss was larger. Blending of PNF-200 into PNF-LT stocks reduced the iso-octane losses while the aromatic constituent losses were still nearly double or more than PNF-200 alone.

P. Touchet of MERDC evaluated several coated fabric (ref. samples as seen in Table 18. One can see that on the combined balance of fuel diffusion rate, tensile strength elongation and torsional stiffness ratio, formulation R211922 was ahead. Unfortunately, it is very difficult to process since it is quite nervy and sticky. R211924 seemed to offer some processability advantages and was examined further.

Table 19 through 24 present formulations which attempted to address the processability problems from various aspects. First this was studied by increased clay or filler loading and then by low molecular weight silicone process aid, HA-2, addition. Also, a fluorosilicone process aid, FSE262U, addition was used. Some slight improvements in release from mill rolls was seen for the low levels of process aid addition.

An alternate approach was considered, namely thermal aging of the polymers. This appeared to offer some improvement in the processability of PNF-LT in the hose building programs previously mentioned. First attempts with this approach are presented in Tables 21 and 22 where a silicone process aid is included in the formulation. Since the diffusion results with these formulations were so poor (i.e., diffusion rates > 0.2 fl. oz./sq. ft. - 24 hours), a second series of compounds without the use of the silicone process aid was initiated as shown in Table 23 and 24.

The stress-strain properties, tensile strength, appeared to improve slightly over the previous "process aid" series. The high filler approach seemed to offer the processability advantage, such as R211924 in Table 11.

Several large mixes were prepared in a Banbury "BR" size mixer with the high filler level loading of formulations similar to R211924 which showed earlier good diffusion rate results. These mixes are presented in Table 25 along with some coagent additions to small samples of each batch. The coagent did increase the modulus, elongation was reduced and diffusion rate for vulcanizates was lower.

Samples of the large batches were prepared (see Table 26) and submitted to Weather-O-Meter aging using the No. 47 cam which gave a period of 18 hours of 102 min. light and 18 min. water spray cycles followed by a period of 6 hours of dark. The retention of tensile properties is quite good with only a 15-20% reduction over 500 hours of total light exposure. The R11988 vulcanizate containing a thermal stabilizer and hence a tan-yellow tint was bleached out after the exposure to light. The R211992 vulcanizate (not containing the stabilizer but additional silane) was light color to start and also lightened under light exposure.

Table 27 shows the stress-strain properties after aging R211988 in test fuel "G" at 160°F. After 14 days the tensile strength and elongation are within specification. After this first initial drop in tensile values, the change to 42 days is very small. After 42 days the tensile strength is within the range set by the specification.

Since the role of metal impurities in contact with jet fuels has an important influence on the oxidative stability of fuels such as JP-4, an analysis was conducted on several polymer samples. Copper has an especially deleterious effect on the storage stability of JP-4. This is reported to occur at low soluble copper content (<0.3 ppm) in fuel and also in contact with copper (ref. 15).

Atomic absorption metal analysis showed the following values for PNF polymer gum prior to any compounding.

| | Atomic Absorption Metal Analysis | | | |
|-----------------|----------------------------------|-----------|-----------|-----------|
| | Parts Per Million (ppm) | | | |
| | <u>Cu</u> | <u>Fe</u> | <u>Ni</u> | <u>Cr</u> |
| PNF Polymer | | | | |
| K18356 (PNF-LT) | 0.01 | 2 | none | 0.1 |
| K19736 PNF-200 | 0.09 | 0.8 | none | 0.07 |
| K19861 | 0.08 | 2 | none | 0.08 |
| K19862 | 0.09 | 2 | none | 0.09 |

One can see the levels to be low for copper.

The filterability of a fuel is affected by the presence of particulate matter in the fuel whether it is dirt, solidified fuel or ice, produced by low temperatures, or insoluble gum caused by aging of the fuel. The presence of insoluble gum aggravates the effect of ice on filter plugging, probably due to the fact that it provides nucleation sites for the growth of ice crystals. While filtration should remove existent gum, the event of by-passing the filters under emergency military situations exists. Therefore, the lowest existent gum values are desired.

A check of fuel contamination of the PNF-200 R211988 and R211992 compounds was conducted. Table 28 compares the values of unwashed existent gum, heptane-washed gum and stored gum residue for these compounds with the values required in MIL T-52573 and that of this contract.

One can see that very little gum is generated in the test fuel by the PNF compounds. The values determined in the tests show PNF materials are well below the required values.

Table 29 presents fuel diffusion rate data for a number of coated fabrics and vulcanizates. For purposes of comparison the fuel diffusion rates of current MIL T-52573C coated fabric was measured with both "Fuel δ " and Type II fuel. One type of material appeared to pass the requirements with 0.037 and 0.05 fl oz/sq ft - 24 hours. The PNF-200 compounds appeared to have diffusion rates which were close to that desired, but depended upon fabric surface treatment.

The lowest diffusion levels were seen with purified PNF-200 polymer and a thick surface treatment to the nylon. This was too rigid to meet flexibility requirements. A lighter coating of fabric treatment looked promising (TX13 and TX14).

The nature of fabric surface treatment was explored further as shown in Table 30 where various treatments were applied to the fabric. The Thixon material showed the best results. Table 31 shows the effects of these treatments upon the breaking strengths of the coated fabrics and the retention of this property with aging in water and test fuel 8. The best breaking strengths were seen with a Fluorel 5150 treatment. However, these showed the greatest loss in water aging. Thixon 300/301 treated fabric had the best overall aging property.

Table 32 presents several mixes with different filling factors in a measuring head mixer. Some exceptionally good tensile strength and elongation values are seen for several mixes where the temperature of the mix was maintained at a low values 97°C or less.

This procedure was used in later Danbury mixes but the tensile strengths were not as high as those realized here.

The adhesive peel strength was examined for coated fabrics which had various surface treatments. Efforts were directed to attempt to attain the adhesive peel strength of about 20 ppi.

The values shown in Tables 33, 34, 35 show Thixon A/B to be the best performing surface treatment. However, the value of about 9 ppi is attained with a heavy coating on the fabric. The breaking strengths are shown in Table 35 and role of temperature of cure indicates that 170°C is better for promotion of breaking strength.

Examination of fracture surfaces of tensile specimens indicated large agglomerates of stabilizer crystals were present. In an attempt to reduce this particle size of the stabilizer it was ball milled with Burgess KE clay for 24 hours and prepared as a masterbatch for the Danbury mixes to follow. See Table 26 for the masterbatch formulation. Later, examination of fracture surfaces of Danbury mixes showed a reduced size but still identifiable particles of stabilizer. This is an area requiring further study in the future.

Since large quantities of compound were needed for calendering, a number of Banbury mixes were made with formulations representing the best combination of properties: processing, diffusion rate and stress-strain. The mixes and their properties are shown in Table 37. Table 30 presents diffusion rates.

Formulations 219601 and 602 were identical except for the PNF-200 polymer batch. This entire mix was blended and labeled as 219601 so that samples could be prepared of coated fabric for shipment. The mix temperature of this series was kept low to achieve higher viscosity and greater shear mixing.

Formulation 219607 represented the best physical property formulation with low diffusion but was difficult to process as a calenderable material. It was used to prepare cement coatings on fabrics before building coated fabrics.

Earlier work with small batches of purified polymer showed some improvement in diffusion rate when compared to normal polymer. Table 38 presents this data. The polymer was purified by making a 14% PNF-200, K19861, solution in methanol, filtering, followed by coagulation in water. Based on these findings, a 9 lb. quantity of polymer was committed to a purification procedure and included in the Banbury mixes.

Formulation 219608 and 219609 were mixed and compared to evaluate the role of an additional purification step on the PNF-200 polymer. Polymer 1508-49 was obtained by dissolving polymer K19861 in methanol, 1 lb/gal, followed by dilution with one gallon of methanol. Centrifugation removed some particulate matter. The cement was coagulated in deionized water, collected and vacuum dried. Only small differences were seen between the two formulations in original properties and even after aging in Fuel S for 14 days at 160°F as seen in Table 39. The diffusion rates for R219608 and R219609 are nearly identical for both vulcanizates and coated fabric.

Table 41 lists the breaking strengths of coated fabrics prepared from the above Banbury mixed stocks with different fabric surface treatments, Thixon 300/301 and Fluorel 5150. The breaking strengths were obtained on water aged fabrics after 7 days at 160°F. Here the stock 219609 with purified polymer shows improved properties compared to 219608. The Fluorel 5150 treated fabrics showed slightly better properties than the Thixon except for the purified polymer stock 219609.

A roll wrap cure procedure was examined to determine if similar physical properties could be obtained to press cured materials. Table 42 shows the results of a calendered length of material wrapped around an aluminum tube interleaved with steel skim for heat transfer and surface finish purposes. Samples were taken every six inches along the length and tested. Comparison is made to press cured material. One can see only a very small difference in properties.

This establishes a production-type of procedure which was used to prepare calendered and cured stock and also coated fabric material. Lengths of seam were also prepared in this manner.

A number of adhesion pads were prepared to establish initial seam adhesion peel strengths. Calendered fabric (uncured) was plied and press cured with a 1" strip of Holland cloth at one end to permit ready gripping in a tensile test machine. Initial strengths of about 9.0 ppi were obtained; however, there was ready migration of the tear to the fabric and a fabric-to-rubber strength of about 6.0 ppi was observed. When additional adhesion promoters were used on fabric and seam, slightly higher values of peel strength were seen as presented in Table 43.

This area of fabric adhesion and seam strength adhesion needs further study and improvement.

2.4 Material Shipments

On February 1, 1978 the contractor acknowledged receipt of:

10 lbs. of PNF-LT K18356
10 lbs. of PNF-200 K19736

as a partial shipment from Contract DAAG46-78-M-1204.

After a decision was reached in a May 24, 1978 meeting with the Contracting Officer's representative, Dr. Robert E. Singler, the shipment of 80 lbs. of PNF-200 was made June 15, 1978 and receipt of

8 lbs. of K19736)
62 lbs. of K19861) PNF-200
10 lbs. of K19862)

was acknowledged.

Also, 40 lbs. of Government Furnished Material as PNF-200 compounded gum elastomer stock (approximately 50% PNF-200) was received by this contractor as 18 lbs. of R211,988 and 22 lbs. of R211992 compounded elastomer stock on October 31, 1978 as per purchase order DAAG46-78-M-1256.

Under this contract shipment of materials to AMMRC included:

20 lbs. K19861 PNF-200 Polymer - 10 October 1978.

Item 0001AB

1. 18 square feet of PNF-200 coated fabric.

| | |
|--------------------------------|---------------------|
| R219601 9-3/4" x 73" x 55 mil | 4.9 sq. ft. |
| R219601 9-3/4" x 72" x 55 mil | 5 |
| R211992 10" x 39" x 50 mil | 2.7 |
| R211992 10" x 43-1/2" x 56 mil | 3.0 |
| R211992 9-1/2" x 59" x 56 mil | 3.9 |
| | <u>19.6</u> sq. ft. |

2. 4 square feet of PNF-200 cured compound.

Press cured.

| | |
|--------------------------------|-----------|
| 8 each 6" x 6" x .075" R211992 | |
| 8 each 6" x 6" x .075" R219601 | 4 sq. ft. |

Wrap cured material.

| | |
|---------------------------------|---------------------|
| 1 each 6" x 48" x .065" R219608 | 3.25 sq. ft. |
| 1 each 6" x 30" x .065" R219601 | |
| | <u>7.25</u> sq. ft. |

3. PNF-200 seam adhesion samples 6 ft. length

10" wide seam x 51" long wrap cured, R219601

5 each 8" x 10" press cured adhesion pads, R211988

1 each 4" x 10" R211988

1 each 4" x 10" R219601

8.25 ft. of seam material

Also shipped were 12 each 6" x 6" x .078" ASTM sheets
of R211988, R211992, R219601 and R219607.

3.0 Conclusions

1. PNF-200 has better fuel diffusion properties than PNF-LT.
2. Diffusion test results indicated that a diffusion barrier layer was not needed.
3. The low temperature flexibility of the low filler content PNF-200 compounds appears adequate to meet service requirements at -70°F.
4. Additional development is required to improve adhesion-to-fabric substrate.
5. Processability was not improved by attempted thermal degradation of the PNF-200 polymer at 300°F. The PNF-200 polymer DSV or ML4-212 did not change very much even after 12 hours aging. (See Table 21)
6. Silicone and fluorosilicone process aids proved unsatisfactory because fuel diffusion rates were adversely affected.
7. The breaking strengths of the coated fabric were less than the original material. Several factors could contribute to this:
a) the fabric may be mechanically damaged in the fabrication process, b) chemically damaged by the fabric surface treatment, and c) the PNF-200 compound may contain small amounts of residual fluoroalcohol (a known solvent for nylon) and contribute to early failure due to stress corrosion cracking.
8. The low temperature flexibility of PNF compounds is quite good. However, the representation of TSR (torsional stiffness ratio) does not adequately reflect the low modulus of rigidity of the PNF vulcanizates and coated fabrics. Values of modulus should also be examined at the low temperatures and not merely the ratio to the room temperature modulus.

4.0 Recommendations

1. Improve fabric-coating adhesion.
2. Consider selection of alternate fabric materials with reduced weight and increased strength.
3. Study development of stronger seam bonds.
4. Consider further processability improvement studies to promote fabric calendering at rapid speeds.

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Glossary

| <u>Item</u> | <u>Description</u> | <u>Source</u> |
|---------------|--|-------------------------------------|
| Burgess KE | Surface treated anhydrous aluminum silicate. | Burgess Pigment Co. |
| Chemlok 607 | Adhesion promoter | Hughson Chemicals, Lord Corporation |
| Chemlok AP134 | Adhesion promoters for fluorocarbon rubber containing silicone, toluene, butyl cellosolve and n-butanol. | Hughson Chemicals, Lord Corporation |
| Chemlok AP133 | | |
| Elastomag 170 | High activity magnesium oxide. | Akron Chemical |
| FEF | Fast extruding furnace black. ASTM classification N550. | |
| Fluorel 5150 | A bonding agent for rubber-to-metal and rubber-to-rubber for fluoroelastomers. | 3M, Commercial Chemicals Div. |
| FSE262U | Fluorosilicone rubber compound (processing aid). | General Electric |
| HA-2 | Silicone rubber compound (processing aid) | Dow Corning |
| Mistron Vapor | Ultrafine magnesium silicate pigment. | Cyprus Ind. Minerals Co. |
| Min-U-Sil | 5 micron ground quartz | Penn Glass Sand Corp. |
| PNF-200 | A phosphonitrilic fluoroelastomer containing pendant fluoroalkoxy groups. | Firestone |
| PNF-LT | A phosphonitrilic fluoroelastomer with a reduced level of fluorine in the polymer. | Firestone |
| phr | per hundred parts by weight rubber. | |
| Silane A151 | Vinyltriethoxysilane | Union Carbide |
| Silane A174 | gamma-methacryloxypropyl-trimethoxysilane | Union Carbide |

Glossary (cont'd)

| <u>Item</u> | <u>Description</u> | <u>Source</u> |
|-------------------|--|---|
| Silane A1100 | gamma-aminopropyltriethoxy-silane | Union Carbide |
| Stabilizer | Zinc II bis(8-oxyquinolate) | Southland |
| TAIC | Triallylisocyanurate | Allied Chemical |
| Test Fuel: | | |
| Type II, TTS-735 | 60V iso-octane 5V benzene 20V toluene 15V xylenes | |
| Test Fuel: | | |
| Type 6 | 60V iso-octane 25V toluene 15V xylenes | |
| Thixon 300/301 | A one coat adhesive for bonding fluorocarbon elastomers to metal. Previously designated as Thixon A/B 273. | Whittaker, Dayton Coatings & Chemicals Division |
| Tullanox 500 | Hydrophobic fumed silica | Tulco |
| Tulcup 40KE | 40% α , α' -bis(t-butylperoxy) diisopropylbenzene | Hercules |
| Water ground Mica | -325 mesh water ground mica. | C. P. Hall |
| Z-6020 | Aminoalkyl functional silane | Dow Corning |
| Zeolox 23-A1100 | Surface treated silica | J. M. Huber |
| Zonyl FSN | A fluoxosurfactant, non-ionic. | DuPont |

TABLE 1. DESIRED PROPERTIES OF CANDIDATE COATING COMPOUNDS

| | | |
|---|---|---------------------|
| Initial | | |
| Tensile strength, psi (min.) | 1,500 | |
| Ultimate elongation, % (min.) | 200 | |
| Properties after immersion in test fluid TT-S-735, Type II at 160°F for 14 days | | |
| Volume change % (max.) | 40 | |
| % Retention of initial tensile strength (min.) | 60 % (or 900 psi) | |
| Properties after immersion in distilled water at 160°F | | |
| Volume change % | 14 days record | 42 days record |
| % retention of initial tensile strength. | 60%(min.) (900 psi) | 50%(min.) (750 psi) |
| Properties after accelerated weathering for 500 hours at 10% elongation (exterior compound). | | |
| | 75%(min.) (1125 psi) | |
| Stiffness at -70°F (after 4 days at -70°F) | | |
| | Maximum of 5 times stiffness at + 73°F. | |
| Value (after 4 days at -70°F) | record | |
| Fuel contamination | | |
| Unwashed existent gum mg/100 ml, max. | 20 | |
| Heptane washed existent gum, mg/100 ml, max. | 5 | |

TABLE 2. DESIRED PROPERTIES OF COATED FABRIC

| | | |
|---|--|---------------------|
| Weight, ounces/sq. yd. | 30-50 | |
| Thickness, mils | record | |
| Type II Fuel diffusion rate fluid ounces per square foot per 24 hours, max. | 0.050 | |
| Tearing strength, lbs. | | |
| Warp, min. | 35 | |
| Fill, min. | 35 | |
| Breaking strength, lbs./in. | | |
| Warp, min. | 400 | |
| Fill, min. | 400 | |
| Puncture resistance, lbs. min. | 110 | |
| Properties after 500 hours accelerated weathering at 5% elongation, initial tensile strength, % retained | | |
| Warp, min. | 80 | |
| Fill, min. | 80 | |
| Low temperature (-70°F, 4 days) Crease resistance | | |
| Appearance after unfolding | No cracking, peeling or delamination. | |
| Fuel diffusion rate, fluid ounces per square foot per 24 hours, max. | 0.050 | |
| Blocking test | Test specimen separates with 5 seconds. | |
| Coating adhesion initial lbs/in.(min.) | 20 | |
| Coating adhesion after immersion in distilled water at 160°F (lbs./in.) | 14 days 10 (min.) | 42 days 5 (min.) |
| Coating adhesion after immersion in Type II fluid at 160°F (lbs./in.) | 14 days 10 (min.) | 42 days 7 (min.) |

TABLE 3. PNF-200 VULCANIZATE AND COATED FABRIC PROPERTIES - SUMMARY

| | LOW FILLER LEVEL FORMULATIONS | HIGH FILLER LEVEL FORMULATIONS |
|---|-------------------------------|--|
| | | |
| Diffusion Rate fl. oz./sq. ft. 24 hrs. | R211922 0.073-0.08 | R211924 (or 988, or 608) 0.01 to 0.11 |
| Coated Fabric Wt. | 37.8 to 55 | 45 to 64 |
| Breaking Strength | 370 ppi | 240-480 ppi |
| ASTM D3388 | | |
| Low Temperature Flexibility G psi, Room Temperature ASTM D 1053 | Vulcanizate 34 psi | Coated Fabric 593 psi |
| | -57°C | TSR -70°F 6.6 |
| T ₅ T ₁₀ T ₁₀₀ | -60°C | TSR -70°F 19.8 |
| | -67°C | |
| Vulcanizate Properties: | 1562 to 1616 | 1309 |
| Tensile Strength | 294 to 217 | 140 |
| Elongation, % | — | 91% (1192 psi) |
| Tensile Strength | 60% (900 psi) | |
| Retention-Fuel Aging | 14 days | |
| Fuel Contamination | 20 mg/100ml | 4.0 to 1.0 |
| Unwashed Existent Gum | 5 mg/100ml | 2.0 to 0.6 |
| Heptane Washed Gum | — | Fair-Good |
| Processability | Poor-Fair | |

TABLE 4. NYLON FABRIC PROPERTIES

Nylon 66

8.25 oz/sq. yd.
2x2 basket weave

Physical Properties:

| | | | |
|-------------------|------|-----|--------|
| Breaking Strength | Warp | 480 | Lbs/in |
| | Fill | 480 | Lbs/in |

| | | | |
|---------------|------|-----|------|
| Tear Strength | Warp | 120 | Lbs. |
| | Fill | 100 | Lbs. |

Finish: scoured and heat set

Yarn Description:

| Type | | | | | |
|------|--------------|-------|------------|-----------|-------|
| Warp | 705 or equal | 840/1 | Denier/ply | Producers | Twist |
| Fill | 705 or equal | 840/1 | Denier/ply | Producers | Twist |

Yarn Construction:

| | | | |
|-------|------|-------|-----------|
| Count | Warp | 35-37 | ends/inch |
| | Fill | 33-35 | ends/inch |

| | |
|--------|------------------------|
| Weight | 8.0 - 8.5 oz./sq. yd. |
| Gauge | 0.0165 - 0.0195 inches |

TABLE 5. POLYMER DATA

| | PNF®-LT | PNF®-200 | | | |
|-------------|--------------------|-------------------|-------------------|-------------------|-------------------|
| K Number | K18356 | K19736 | K19861 | K19862 | K19730 |
| DSV (dl/g.) | 2.77 | 2.62 | 2.3 | 2.26 | 2.29 |
| Gel (%) | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Na (wt. %) | 0.013 | 0.0084 | 0.014 | 0.012 | 0.026 |
| Cl (wt. %) | 0.074 | 0.027 | 0.022 | 0.03 | 0.094 |
| Tg °C | -76.5°C | -65°C | -65°C | -66°C | -71.5°C |
| M_w | 13.8×10^6 | 9.6×10^6 | 7.7×10^6 | 8.0×10^6 | 7.6×10^6 |
| ML-4, 212°F | 17.0 | 17.6 | 14.9 | 16.7 | 14.5 |

Amount of
GFM (Government Furnished Material)
obtained: 10 lbs. (Received 1 Feb. 1978)
(Received 15 June 1978) 8 lbs. 10 lbs.
(Received 26 Oct. 1978)

22.5 lbs. polymer
as 40 lbs.
compounded stock
(18 lbs. R211,988
22 lbs. R211,992)

* Gel Permeation Chromatography data based on PNF standards (MW used in calibration).

**20 lbs. K19861 shipped to R. Singler, AMRC 10 Oct. 1978

TABLE 6. PNF-200 POLYMER COMPARISON VIA COMPOUNDING

Formulation A. 15 phr Tullanox 500, 5 Zeolex 23, 6 Elastomag 170, 2 stabilizer, 2.5 Silane AL74, 0.4 Vulcup 40KE

Formulation B. 70 phr Burgess KE, 15 Mistron Vapor, 10 Zeolex 23, 8 Elastomag 170, 2 stabilizer, 0.5 Vulcup 40KE

| | | | | |
|----------|---------------|--------|---------------|--------|
| | Formulation A | | Formulation B | |
| Polymer: | K19736 | K19861 | K19736 | K19861 |

Monsanto Rheometer Data. T = 340°F, 1° ARC, 100 RPM Mini Die.

| | | | | | | |
|-------------------|---------|------|------|------|------|------|
| | R211944 | -945 | -946 | -947 | -948 | -949 |
| Scorch Time | 1.8 | 1.7 | 1.7 | 3.2 | 3.3 | 3.6 |
| Opt. Cure TC(90) | 17.3 | 14.5 | 15.3 | 21.0 | 23.9 | 25.0 |
| Min. Torque | 6.6 | 6.5 | 5.9 | 13.5 | 12.1 | 12.1 |
| Torque @ 90% Cure | 17.0 | 16.8 | 16.6 | 26.6 | 23.6 | 20.9 |
| Max. Torque | 18.1 | 17.9 | 17.8 | 28.0 | 24.9 | 21.9 |
| Cure Rate Index | 6.5 | 7.8 | 7.3 | 5.6 | 4.9 | 4.7 |

Cure 30 Min. @ 340°F:

Ring Tensile Data. Post Cure 4 hrs. @ 350°F (0 indicates no post cure.)

| | | | | | | | | | | |
|--------------|------|------|------|------|------|------|------|------|------|------|
| | 0 | X | 0 | X | 0 | X | 0 | X | 0 | X |
| Tensile Str. | 1566 | 1797 | 1813 | 1742 | 1390 | 1813 | 1210 | 1102 | 1124 | 1085 |
| (psi) | | | | | | | | | | |
| 50% Modulus | 149 | 221 | 139 | 144 | 141 | 148 | 676 | 503 | 526 | 570 |
| 100% Modulus | 450 | 644 | 369 | 346 | 364 | 375 | 1209 | 1070 | 1007 | 1054 |
| 200% Modulus | 1461 | - | 1305 | 1128 | 1200 | 1109 | - | 105 | 122 | 109 |
| Elongation | 202 | 189 | 239 | 257 | 219 | 272 | 103 | 105 | 122 | 109 |
| Shore A | 50 | 55 | 55 | 55 | 55 | 55 | 75 | 70 | 70 | 70 |

| | | | |
|----------------|------|------|------|
| Diffusion Rate | 0.37 | 0.33 | 0.32 |
| fl. oz/sq. ft. | | | |
| 24 hrs. | | | |

| | | | |
|----------------|----|----|----|
| Wt. oz/sq. yd. | 42 | 41 | 44 |
| Coated Fabric | | | |

TABLE 7. PNF-200 FORMULATIONS

| R-Number 211 PNF-200: K19736 | <u>-901</u> | <u>-904</u> | <u>-906</u> | <u>-911</u> | <u>-920(904)</u> | <u>-922(901)</u> |
|---|-------------|-------------|-------------|-------------|------------------|------------------|
| | 100 | 100 | 100 | 100 | 100 | 100 |
| Tullanox 500 | 15 | - | - | 7 | - | 15 |
| Burgess KE | - | 15 | - | - | 15 | - |
| Nulok 321SP | - | - | 15 | - | - | - |
| FEF | - | - | - | 8 | - | - |
| Zeolex 23 | 5 | 5 | 5 | 5 | 5 | 5 |
| Elastomag 170 | 6 | 6 | 6 | 6 | 6 | 6 |
| Stabilizer | 2 | 2 | 2 | 2 | 2 | 2 |
| Silane A151 | 2 | 2 | 2 | 2 | - | - |
| Silane A174 | - | - | - | - | 2.5 | 2.5 |
| Vulcup 40KE | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 |
| Cure 30 Min. @ 340°F | | | | | | |
| Tensile Str. | 681 | 1036 | 968 | 1322 | 1189 | 1616 |
| 50% Modulus | - | - | 94 | 108 | 111 | 175 |
| 100% Modulus | 341 | 311 | 343 | 374 | 473 | 564 |
| 200% Modulus | - | - | 891 | 1203 | - | 1478 |
| Elongation, % | 160 | 176 | 196 | 210 | 135 | 217 |
| Shore A | 50 | 40 | 45 | 50 | 40 | 60 |
| Specific Grav. | 1.8 | 1.8 | 1.9 | 1.9 | 2.0 | 1.8 |
| G, psi @ 23°C | 34.3 | 37.8 | 42.4 | 64 | | |
| T ₂ , °C | -45.9 | -32.8 | -39.5 | -34.3 | | |
| T ₅ , °C | -52.9 | -42.1 | -46 | -40.8 | | |
| T ₁₀ , °C | -58.5 | -47.0 | -48 | -47.6 | | |
| T ₁₀₀ , °C | -67.1 | -60.1 | -55 | -55 | | |
| Diffusion Rate fl.oz./sq.ft. - 24 hrs. | | | | 0.18 | | |
| 72 hour R.T. Type II Fuel Immersion: | | | | | | |
| W % Gain | 9.7 | 9.7 | 8.0 | 1.4 | 0 | 1.4 |
| V % Gain | 29.1 | 38.5 | 5.6 | 8.3 | 9.5 | 6.8 |

TABLE 8. PNF-LC FORMULATIONS

| R-Number 211 | -902 | -903 | -905 | -909 | -918 (903) | -921 (905) | -923 (902) |
|---------------|------|------|------|------|---------------|---------------|---------------|
| PNF-IT: | | | | | | | |
| K17356 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Tullanox 500 | - | 15 | - | 7 | 15 | - | - |
| Burgess KE | 15 | - | - | - | - | - | 15 |
| Nulox 321SP | - | - | 15 | - | - | 15 | - |
| FEF | - | - | - | 8 | - | - | - |
| Zeolex 23 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Elastomag 170 | 6 | 6 | 6 | 6 | 6 | 6 | 6 |
| Stabilizer | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| Silane A151 | 2 | 2 | 2 | 2 | - | - | - |
| Silane A174 | - | - | - | - | 2.5 | - | - |
| Vulcup 40KE | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 |

Cure 30 min @ 340°F

| | | | | | | | |
|-----------------------|-------|-------|-------|-------|-----|-----|-----|
| Tensile Strength | 1488 | 679 | 802 | 1002 | 867 | 662 | 709 |
| 50% Modulus | - | - | - | 102 | 230 | 90 | 80 |
| 100% Modulus | 337 | 331 | 342 | 344 | 604 | 367 | 336 |
| 200% Modulus | 1282 | - | - | 974 | - | - | - |
| Elongation, % | 213 | 155 | 163 | 204 | 134 | 140 | 147 |
| Shore A | 45 | 45 | 45 | 50 | 60 | 50 | 40 |
| Specific Gravity | 1.8 | 1.9 | 2.0 | 1.9 | 1.7 | 1.8 | 1.8 |
| G, psi @ 23°C | 85.6 | 33.9 | 38.8 | 65.8 | | | |
| T ₂ , °C | -28.6 | -45.3 | -42.4 | -44.9 | | | |
| T ₅ , °C | -42.7 | -57.0 | -56.5 | -53.9 | | | |
| T ₁₀ , °C | -48.2 | -60.2 | -60.9 | -55.4 | | | |
| T ₁₀₀ , °C | -59.4 | -67.0 | -69.8 | -65.2 | | | |

Diffusion Rate
fl.oz./sq.ft. - 24 hrs. 0.38 0.74 0.46

72 hrs. R.T. Type II Fuel Immersion:

| | | | | | | | |
|----------|------|------|------|------|------|------|------|
| W % Gain | 7.5 | 13.2 | 0 | 12.2 | 13.2 | 9.9 | 8.8 |
| V % Gain | 12.9 | 33.8 | 17.4 | 32.8 | 33.8 | 32.9 | 29.1 |

**TABLE 9. PNF-200 FORMULATIONS WITH INCREASING
NON-REINFORCING FILLER LOADING**

| R-Number 211 | <u>-912</u> | <u>-913</u> | <u>-914</u> |
|---|-------------|-------------|-------------|
| PNF-200 K19736 | 100 | 100 | 100 |
| Nulok 321SP | 20 | 20 | 20 |
| Min-U-Sil | 10 | 20 | 30 |
| Zeolex 23-A1100 | 10 | 10 | 10 |
| Elastomag 170 | 6 | 6 | 6 |
| Stabilizer | 2 | 2 | 2 |
| Silane A174 | 2 | 2 | 2 |
| Vulcup 40KE | 0.4 | 0.4 | 0.4 |
| Cure 30 min. at 340°F | | | |
| Tensile Strength, psi | 1250 | 1458 | 1482 |
| 50% Modulus, psi | 167 | 109 | 221 |
| 100% Modulus, psi | 734 | 548 | 970 |
| Elongation, % | 138 | 167 | 131 |
| Shore A | 55 | 60 | 60 |
| Specific Gravity | 2.0 | 2.0 | 2.0 |
| Diffusion Rate fl.oz./sq.ft. - 24 hrs. | | 0.13 | 0.11 |
| 72 hours R.T. Type II Fuel Immersion: | | | |
| W % gain | 1.3 | 1.3 | 0 |
| V % gain | 2.8 | 6.4 | 5.1 |

TABLE 10. PNF-LT/ PNF-200 BLENDS

| R-Number 211 | <u>-915</u> | <u>-916</u> | <u>-917</u> |
|--|-------------|-------------|-------------|
| PNF-200 K19736 | 66.6 | 66.6 | 66.6 |
| PNF-LT K18356 | 33.4 | 33.4 | 33.4 |
| Nulok 321SP | 15 | - | - |
| Burgess KE | - | 15 | - |
| Tullanox 500 | - | - | 15 |
| Zeolex 23 | 10 | 10 | 10 |
| Elastomag | 6 | 6 | 6 |
| Stabilizer | 2 | 2 | 2 |
| Silane Al7 ⁴ | 2 | 2 | 2 |
| Vulcup 40KE | 0.4 | 0.4 | 0.4 |
| Cure 30 min. @ 340°F | | | |
| Tensile Strength | 879 | 882 | 1274 |
| 50% Modulus | 148 | 122 | 277 |
| 100% Modulus | 510 | 489 | 687 |
| Elongation values | 149 | 152 | 163 |
| Shore A | 55 | 55 | 65 |
| Specific Gravity | 1.9 | 1.9 | 1.9 |
| Diffusion Rate fl. oz/sq. ft.- 24 hr. | 0.27 | 0.23 | 0.23 |
| 72 hours R.T. Type II Fuel Immersion: | | | |
| W % gain | 1.3 | 0.7 | 1.4 |
| V % gain | 11.6 | 15.5 | 10.7 |

TABLE 11. PNF-200 FORMULATIONS WITH HIGH FILLER LOADINGS

| R-Number 211 | <u>-924</u> | <u>-925</u> | <u>-926</u> | <u>-927</u> | <u>-928</u> | <u>-929</u> |
|---------------------------------------|-------------|-------------|-------------|-------------|-------------|-------------|
| PNF-200 | | | | | | |
| K19736 | 100 | 100 | 100 | 100 | 100 | 100 |
| Burgess KE | 60 | 45 | 30 | - | - | - |
| Mistron Vapor | - | - | - | 60 | 45 | 30 |
| Zeolux 23-A1100 | 10 | 10 | 10 | 10 | 10 | 10 |
| Stabilizer | 2 | 2 | 2 | 2 | 2 | 2 |
| Elastomag 170 | 8 | 8 | 8 | 8 | 8 | 8 |
| Vulcup 40KE | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 |
| Cure 30 min. @ 340°F | | | | | | |
| Tensile Strength | 1464 | 1163 | 1179 | 1224 | 1410 | 1405 |
| 50% Modulus | 467 | 125 | 155 | 650 | 568 | 400 |
| 100% Modulus | 1362 | 663 | 654 | 1219 | 1267 | 1013 |
| 200% Modulus | - | - | - | - | - | - |
| Elongation, % | 110 | 150 | 168 | 102 | 118 | 153 |
| Shore A | 65 | 60 | 55 | 70 | 65 | 65 |
| Specific Gravity | 1.9 | 1.8 | 1.9 | 1.9 | 1.9 | 1.7 |
| Diffusion rate | 0.09 | 0.18 | 0.19 | 0.14 | 0.16 | 0.24 |
| fl. oz/sq. ft.- 24 hours. | | | | | | |
| 72 hours R.T. Type II Fuel Immersion: | | | | | | |
| W % gain | 17.8 | 18.2 | 13.5 | 13.5 | 13.0 | 20.7 |
| V % gain | 14.4 | 7.06 | 27.6 | 6.9 | 12.9 | 14.5 |

TABLE 12. HIGH CLAY AND TALC LOADING OF PNF-LT AND PNF-200

| R-Number 211- | <u>-936</u> | <u>-937</u> | <u>-938</u> | <u>-939</u> | <u>-940</u> | <u>-941</u> |
|---------------------------------------|-------------|-------------|-------------------------|-------------|-------------|-------------|
| PNF-200 K19736 | 100 | - | 100 | 100 | - | - |
| PNF-LT K18356 | - | 100 | - | - | 100 | 100 |
| Burgess KE | 70 | 70 | 60 | 80 | 60 | 80 |
| Mistron Vapor | 15 | 15 | 10 | 10 | 10 | 10 |
| Zeolex 23-A1100 | 10 | 10 | 10 | 10 | 10 | 10 |
| Stabilizer | 2 | 2 | 2 | 2 | 2 | 2 |
| Elastomag 170 | 8 | 8 | 8 | 8 | 8 | 8 |
| Vulcup 40KE | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 |
| Cure 30 Min. 340°F | | | | | | |
| Tensile strength | 1213 | 867 | 1407 | 1251 | 828 | 810 |
| 50% Modulus | 712 | 873 | 664 | 756 | 639 | 710 |
| 100% Modulus | 1203 | - | 1323 | - | - | - |
| Elongation | 102 | 55 | 107 | 86 | 70 | 65 |
| Shore A | 75 | - | 70 | 75 | - | - |
| Specific Gravity | 1.9 | 1.8 | 1.8 | 1.9 | 1.7 | 1.8 |
| | | | | | | |
| Diffusion Rate | 0.07, 0.10 | | .084, 0.10, 0.075, 0.09 | | | |
| fl.oz/sq.ft.-24 hrs | | | | | | |
| Fabric Wt. | 83, 54 | | 78, 55 | | 78, 56 | |
| 72 hours R.T. Type II Fuel Immersion: | | | | | | |
| Wt. % gain | 11.5 | 17.3 | 13.0 | 13.5 | 21.7 | 25 |
| Vol. % gain | 7.8 | 20.5 | 10.3 | 5.7 | 14.7 | 21.4 |

TABLE 13. FUEL DIFFUSION RATE OF COATED FABRICS - PNF-LT AND PNF-LT/200 BLENDS

Test Fuel: TT-S-735 Type II
(60V Iso-octane, 5V Benzene 20V Toluene, 15V Xylenes)

| | Fuel Diffusion Rate | Coated Fabric Weight | Tensile Strength MPa(psi) | Elongation % |
|-------------------------|-----------------------------------|----------------------------|---------------------------------|-----------------|
| Specification | 0.05 fl. oz/sq.ft. -24 hrs. | 40-48 oz./ sq/yd. | 10.3 (1500) | 200% |
| <u>PNF-LT</u> | | | | |
| R211,909 | 0.38 | 39.0 | 6.91 (1002) | 204 |
| R211,918 | 0.74* | 45.9 | 5.98 (867) | 134 |
| R211,923 | 0.46 | 44.1 | 4.89 (709) | 147 |
| <u>PNF-LT/PNF-200</u> | | | | |
| R211,910 | 0.21 | 37.5 | 7.90 (1146) | 211 |
| R211,919 | 0.30 | 41.7 | 6.77 (982) | 168 |
| <u>PNF-LT/2 PNF-200</u> | | | | |
| R211,915 | 0.27 | 34.9 | 6.06 (879) | 149 |
| R211,916 | 0.23 | 40.5 | 6.08 (882) | 152 |
| R211,917 | 0.23 | 39.2 | 8.78 (1274) | 168 |

* Diffusion sample had a flaw on interior surface.
Diffusion rate may not be representative.

TABLE 14. FUEL DIFFUSION RATE OF COATED FABRICS OF PNF-200

Test Fuel: TTS-735 Type II
(60V Iso-octane, 5V Benzene, 20V Toluene, 15V Xylene)

| | Fuel Diffusion Rate | Coated Fabric Weight | Vulcanizate | |
|----------------|-----------------------------------|----------------------------|----------------------------------|-----------------|
| | | | Tensile Strength MPa (psi) | Elongation % |
| Specification: | 0.05 fl. oz/sq.ft. -24 hrs. | 40-48 oz./ sq.yd. | 10.3 (1500) | 200% |
| <u>PNF-200</u> | | | | |
| R211,911 | 0.18 | 40.7 | 9.11 (1322) | 210 |
| R211,913 | 0.13 | 45.1 | 9.25 (1342) | 142 |
| R211,914 | 0.11 | 38.9 | 9.65 (1399) | 122 |
| R211,920 | 0.145 | 42.3 | 8.20 (1189) | 135 |
| R211,922 | 0.073 | 37.3 | 11.1 (1616) | 217 |
| R211,924 | A. 0.09 B. 0.08 | 56.5 46.8 | 10.1 (1464) | 110 |
| R211,924R | A. 0.096 B. 0.10 | 64 54 | 10.1 (1464) | 110 |
| R211,925 | 0.18 | 48 | 8.02 (1163) | 150 |
| R211,926 | 0.19 | 45 | 8.13 (1179) | 168 |
| R211,924 | 0.02 (Arctic Diesel Fuel) | | | |

TABLE 15. FUEL DIFFUSION RATE OF COATED FABRICS

Test Fuel: TTS-735 Type II
(60V Iso-octane, 5V Benzene, 20V Toluene, 15V Xylene)

| | Fuel Diffusion Rate | Coated Fabric Weight | Vulcanizate | |
|----------------|------------------------------------|----------------------------|----------------------------------|-----------------|
| | | | Tensile Strength MPa (psi) | Elongation % |
| Specification: | 0.05 fl. oz/sq.ft. - 24 hrs. | 40-48 oz./ sq.yd. | 10.3 (1500) | 200% |
| R211,927 | 0.14 | 48 | 8.44 (1224) | 98 |
| R211,928 | 0.16 | 42 | 8.26 (1198) | 101 |
| R211,929 | 0.24 | 34 | 9.46 (1373) | 144 |
| R211,936 | A. 0.07 B. 0.10 | 83 54 | 8.36 (1213) | 102 |
| R211,938 | A. 0.084 B. 0.10 | 78 55 | 9.70 (1407) | 107 |
| R211,939 | A. 0.075 B. 0.09 | 78 56 | 8.19 (1189) | 86 |
| R211,94 | 0.17 | 42 | 6.02 (874) | 183 |

TABLE 16. DIFFUSION TEST FUEL SAMPLE ANALYSIS

COMPONENT WEIGHT PER CENT LOSSES OVER TEST DURATION

Test Fuel: TT-S-735 Type II

| <u>PNF-LT</u> | <u>Diffus- ion Rate fl.oz/ sq.ft. (24 hrs)</u> | <u>Coated Fabric oz./sq. yd.</u> | <u>Test Dura- tion Days</u> | <u>Iso- Octane</u> | <u>Ben- zene</u> | <u>Tol- uene</u> | <u>Xylenes</u> |
|-------------------------------|--|--|---|------------------------|----------------------|----------------------|----------------|
| R211,909 | 0.38 | 39 | 23 | 7.6 | 41 | 26.9 | 17.7 |
| R211,918* | 0.74 | 45.9 | 38 | 50.2 | 95 | 90.4 | 78.1 |
| R211,923 | 0.46 | 44.1 | 28 | 10.2 | 66.3 | 46.5 | 31.1 |
| <u>PNF-LT/PNF-200 BLEND</u> | | | | | | | |
| R211,910 | 0.21 | 37.6 | 39 | 2.4 | 49 | 31.4 | 21.9 |
| <u>PNF-LT/2 PNF-200 BLEND</u> | | | | | | | |
| R211,916 | 0.23 | 40.5 | 39 | 1.8 | 55 | 36.4 | 23.8 |
| R211,917 | 0.23 | 39.2 | 23 | 8.4 | 35.5 | 18.4 | 12.0 |

*Sample had a flaw on interior surface coating after removal from diffusion test cup. Diffusion result may not be representative.

TABLE 17. DIFFUSION TEST FUEL SAMPLE ANALYSIS

COMPONENT WEIGHT PER CENT LOSSES OVER TEST DURATION

Test Fuel: TT-S-735 Type II

| <u>PNF-200</u> | <u>Diffus- ion Rate fl.oz./ sq.ft. (25 hrs)</u> | <u>Coated Fabric oz/sq. yd.</u> | <u>Test Dura- tion Days</u> | <u>Iso- Octane</u> | <u>Ben- zene</u> | <u>Tol- uene</u> | <u>Xylenes</u> |
|-----------------|---|---|---|------------------------|----------------------|----------------------|----------------|
| R211,913 | 0.13 | 45.1 | 23 days | 0 | 17.0 | 9.1 | 6.1 |
| R211,914 | 0.11 | 38.9 | 23 | 3.8 | 18.9 | 13.3 | 11.1 |
| R211,920 | 0.15 | 42.3 | 38 | 1.3 | 40.0 | 21.2 | 8.9 |
| R211,922 | 0.07 | 37.8 | 28 | 0 | 15. | 7.1 | 12.2 |
| R211,924 A.O.09 | A.O.09 | 56.5 | 38 | 0.6 | 19.3 | 12.4 | 5.8 |
| R211,924 B.O.08 | B.O.08 | 46.8 | 28 | 0 | 16.1 | 8.5 | 7.5 |

TABLE 18. LOW TEMPERATURE FLEXIBILITY PNF[®] COATED FABRIC*

| | | | | | | |
|--|------|------|------|------|-------|-------|
| R-Number 211, | 913 | 914 | 917 | 920 | 922 | 924 |
| Room Temp. G, psi | 713 | 740 | 1027 | 1018 | 1275 | 593 |
| 7 days @ -40°F | | | | | | |
| TSR | 2.9 | 2.5 | 3.0 | 2.0 | 2.3 | 2.2 |
| G, psi | 2100 | 1860 | 3040 | 2040 | 2910 | 1300 |
| 7 days at -70°F | | | | | | |
| TSR | 9.7 | 9.3 | 6.4 | 4.5 | 6.6 | 19.8 |
| G, psi | 6900 | 6860 | 6570 | 4550 | 8440 | 11730 |
| Fuel Diffusion Rate Fl. oz/ft ² -24 hrs. | 0.13 | 0.11 | 0.23 | 0.15 | 0.073 | 0.08 |
| Oz/ Yd ² | 45.1 | 38.9 | 39.2 | 42.3 | 37.3 | 46.8 |
| VULCANIZATE PROPERTIES: | | | | | | |
| Tensile Strength | 1458 | 1482 | 1274 | 1189 | 1616 | 1464 |
| Elongation | 167 | 131 | 168 | 135 | 217 | 110 |
| FORMULATION | | | | | | |
| PNF-200 | 100 | 100 | 66.6 | 100 | 100 | 100 |
| PNF-LT | -- | -- | 33.4 | -- | -- | -- |
| Burgess KE | -- | -- | -- | 15 | -- | 60 |
| Nulok 321 SP | 20 | 20 | -- | -- | -- | -- |
| Min-U-Sil | 20 | 30 | -- | -- | -- | -- |
| Tullanox 500 | -- | -- | 15 | -- | 15 | -- |
| Zeolex 23-Al100 | 10 | 10 | 10 | 5 | 5 | 10 |
| Elastomag 170 | 6 | 6 | 6 | 6 | 6 | 8 |
| Stabilizer | 2 | 2 | 2 | 2 | 2 | 2 |
| Silane Al74 | 2 | 2 | 2 | 2.5 | 2.5 | -- |
| Vulcup 40 KE | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 | 0.5 |

*Low temperature flexibility measurements obtained by
P. Touchet, MERDC .

TABLE 19. PNF-200 PROCESSING STUDY-SILICONE ADDITIONS

| R-Number 211- | <u>-950</u> | <u>-951</u> | <u>-952</u> | <u>-953</u> | <u>-954</u> | <u>-955</u> |
|-----------------|-------------|-------------|-------------|-------------|-------------|-------------|
| PNF-200 K19736 | 100 | 100 | 100 | 100 | 100 | 100 |
| HA-2 | 2 | 2 | 2 | 4 | 4 | 4 |
| Tullanox 500 | 20 | 10 | 10 | 20 | 10 | 10 |
| Burgess KE | - | 10 | 10 | - | 10 | 10 |
| Mistron Vapor | - | 5 | 10 | - | 5 | 10 |
| Min-U-Sil | 10 | 10 | 10 | 10 | 10 | 10 |
| Zeolex 23 A1100 | 5 | 5 | 5 | 5 | 5 | 5 |
| Elastomag 170 | 8 | 8 | 8 | 8 | 8 | 8 |
| Silane A174 | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 |
| Vulcup 40KE | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 |

Cure 30 min. 340°F Post Cure 4 hours @ 350°F

| | | | | | | |
|------------------|------|------|------|------|------|------|
| Tensile Strength | 1605 | 1101 | 1115 | 1054 | 1104 | 1231 |
| 50% Modulus | 226 | 144 | 186 | 234 | 176 | 258 |
| 100% Modulus | 558 | 578 | 636 | 594 | 595 | 820 |
| 200% Modulus | 1424 | - | - | - | - | - |
| Elongation, % | 222 | 153 | 155 | 151 | 157 | 145 |
| Shore A | 65 | 60 | 60 | 65 | 60 | 65 |

Mill Processability: Ability to release from mill roll as thin (0.15") sheet.

| | | | | | | |
|-------------------------|------|------|------|------|------|------|
| | Fair | Good | Good | Poor | Fair | Fair |
| Diffusion Rate "Fuel S" | | | 0.25 | | | 0.29 |
| fl oz/sq ft. - 24 hrs. | | | | | | |

| | | | | | | |
|---------------------------------|--|--|----|--|--|----|
| Coated Fabric weight oz/sq. yd. | | | 45 | | | 50 |
|---------------------------------|--|--|----|--|--|----|

TABLE 20. PNF-200 PROCESSING STUDY-FLUOROSILICONE ADDITIONS

| R-Number 211 | <u>-956</u> | <u>-957</u> | <u>-958</u> | <u>-959</u> | <u>-960</u> | <u>-961</u> |
|-----------------|-------------|-------------|-------------|-------------|-------------|-------------|
| PNF-200 K19736 | 100 | 100 | 100 | 100 | 100 | 100 |
| FSE2620U | 2 | 2 | 2 | 4 | 4 | 4 |
| Tullanox 500 | 20 | 10 | 10 | 20 | 10 | 10 |
| Burgess KE | - | 10 | 10 | - | 10 | 10 |
| Mistron Vapor | - | 5 | 10 | - | 5 | 10 |
| Min-U-Sil | 10 | 10 | 10 | 10 | 10 | 10 |
| Zeoole 23 A1100 | 5 | 5 | 5 | 5 | 5 | 5 |
| Elastomag 170 | 8 | 8 | 8 | 8 | 8 | 8 |
| Silane A174 | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 |
| Vulcup 40KE | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 |

Cure 30 Min. 340°F Post Cure 4 hours @ 350°F

| | | | | | | |
|------------------|------|------|------|-----|-----|------|
| Tensile Strength | 1314 | 1173 | 1150 | 733 | 974 | 1155 |
| 50% Modulus | 202 | 209 | 265 | 242 | 173 | 262 |
| 100% Modulus | 499 | 733 | 809 | 578 | 573 | 793 |
| 200% Modulus | 1195 | - | - | - | - | - |
| Elongation, % | 221 | 142 | 134 | 122 | 144 | 137 |
| Shore A | 60 | 60 | 60 | 60 | 50 | 60 |

Mill Processability: Ability to release from mill roll as thin
(.015" sheet). Fair Good Good Fair Good Poor

| | | | | | | |
|--------------------------|--|--|-----|--|--|-----|
| Diffusion Rate "Fuel S" | | | | | | |
| fl. oz/sq. ft. - 24 hrs. | | | .20 | | | .23 |
| Coated Fabric, Weight | | | | | | |
| oz/sq. yd. | | | 48 | | | 43 |

TABLE 21. PNE-200 PROCESSING STUDY - THERMALLY AGED POLYMER

| R-Number 211, Polymer DSV (al/g) PNE-200 | 962 2.14 K19861 | 963 2.22 K19861A4* | 964 2.12 K19861A6 | 965 2.03 K19861A8 | 966 2.06 K19861A10 | 967 2.03 K19861A12 |
|--|------------------------|--------------------------|-------------------------|-------------------------|--------------------------|--------------------------|
| Tullanox 500 | 20* | | | | | |
| Min-U-Sil | 10 | | | | | |
| HA-2 | 2 | | | | | |
| Zeolox 23-A1100 | 5 | | | | | |
| Elastomag 170 | 8 | | | | | |
| Silane A174 | 2.5 | | | | | |
| Vulcup 40KE | C.4 | | | | | |
| f = 0.65 | Mix to 30,000 M-gm-min | total integrated torque | | | | |
| Polymer Torque | 600 | 600 | 500 | 500 | 500 | 500 |
| Peak Torque | 3200 | 3450 | 3400 | 2000 | 3600 | 3650 |
| Plateau Torque | 1000 | 1400 | 1400 | 1200 | 1270 | 1330 |
| Max. Temp. °C | 73° | 82° | 79° | 84° | 92° | 88° |
| Tensile Strength | 1100 | 1010 | 1134 | 1033 | 1238 | 1158 |
| 50% Modulus | 260 | 215 | 241 | 232 | 294 | 259 |
| 100% Modulus | 543 | 481 | 492 | 487 | 599 | 508 |
| 200% Modulus | -- | -- | 1151 | -- | -- | 1090 |
| Elongation, % | 182 | 177 | 202 | 186 | 187 | 211 |
| Shore A | 65 | 65 | 65 | 65 | 65 | 65 |
| Fuel's | | | | | | |
| Diffusion | 0.30 | 0.32 | | 0.31 | | 0.27 |
| Rate fl. oz. sq. ft. - 24 hrs. | | | | | | |
| Coated Fabric | 45 | 48 | | | | |
| Weight oz/sq. yd. | | | | 46 | | 45 |

Same as -950
Except Silane added to mix

*"A4" indicates polymer aged 4 hours in air at 300°F.

TABLE 22. PNF-200 THERMALLY AGED POLYMER PROCESSING STUDY

| R-Number: 211, | 968 | 969 | 970 | 971 | 972 | 973 |
|---|--------|-----------|----------|--------|----------|--------|
| Polymer DSV (dl/g) | 2.14 | 2.05 | 1.86 | 2.57 | 2.45 | 2.4 |
| PNF-200 | K19862 | K19862A4* | K19862A8 | K19736 | K19736A4 | K19736 |
| Tullanox 500 | 20 | | | | | |
| Min-U-Sil | 10 | | | | | |
| HA-2 | 2 | | | | | |
| Zeolex 23-A1100 | 5 | | | | | |
| Elastomag 170 | 8 | | | | | |
| Silane A174 | 2.5 | | | | | |
| Vulcan 40KE | 0.4 | | | | | |
| f=0.65 | | | | | | |
| Polymer Torque | 580 | 600 | 600 | 750 | 750 | 600 |
| Peak Torque | 3800 | 3550 | 4200 | 3500 | 3600 | 3700 |
| Plateau Torque | 1580 | 1500 | 1650 | 1646 | 1638 | 1600 |
| Max. Temp. °C | 89 | 98 | 84 | 92 | 100 | 98 |
| Integrated Torque 20,000 Meter gram minutes | | | | | | |
| Tensile Strength | 1120 | 1276 | 1333 | 1255 | 1098 | 1251 |
| 50% Modulus | 246 | 241 | 324 | 316 | 350 | 387 |
| 100% Modulus | 522 | 536 | 636 | 616 | 661 | 725 |
| 200% Modulus | -- | 1185 | -- | -- | -- | -- |
| Elongation % | 172 | 216 | 198 | 199 | 167 | 158 |
| Shore A | 65 | 60 | 70 | 70 | 70 | 70 |
| Diffusion Rate - Fuel δ | | | | | | |
| fl. oz./sq. ft.-24 | | | | 0.28 | 0.27 | 0.33 |
| Coated Fabric | | | | | | |
| Weight oz./sq. yd. | | | | 51 | 54 | 51 |

*"A4" indicates polymer aged 4 hrs. in air @ 300°F.

TABLE 23. PNF-200 THERMALLY AGED POLYMER PROCESSING STUDY

| R-Number 211, | 976 | 977 | 978 | 979 | 980 | 981 |
|------------------|--------|-----------|----------|----------|-----------|----------|
| PNF-200 | K19861 | K19861A4* | K19861A6 | K19861A8 | K19861A10 | K19861A1 |
| Tullenox 500 | 20 | } | | | | |
| Min-U-Sil | 10 | | | | | |
| Zeolex 25-Al1CO | 5 | | | | | |
| Elastomag 170 | 8 | | | | | |
| Silane Al74 | 2.5 | | | | | |
| Vulcup 40KE | 0.4 | | | | | |
| f = .65 | | | | | | |
| Tensile Str | 1408 | 1536 | 1552 | 1552 | 1408 | 1439 |
| 50% Mod | 202 | 200 | 212 | 290 | 294 | 204 |
| 100% Mod | 530 | 555 | 611 | 719 | 711 | 596 |
| 200% Mod | 1403 | 1420 | 1563 | 1556 | -- | -- |
| Elongation % | 210 | 217 | 199 | 200 | 184 | 193 |
| Shore A | 60 | 60 | 60 | 65 | 65 | 60 |
| Specific Gravity | 1.95 | 1.92 | 1.92 | 1.92 | 1.96 | 1.92 |

*"A4" indicates polymer aged 4 hrs. @ 300°F.

TABLE 24. PNF-200 THERMALLY AGED POLYMER PROCESSING STUDY

| R-Number 211, | 982 | 983 | 984 | 985 | 986 | 987 |
|------------------|--------|-----------|----------|--------|----------|----------|
| PNF-200 | K19862 | K19862A4* | K19862A8 | K19736 | K19736A4 | K19736A8 |
| Tullamox-500 | 20 | | | | | |
| Min-U-Sil | 10 | | | | | |
| Z-olex 23-A110 | 5 | | | | | |
| Elastom 170 | 3 | | | | | |
| Silone A174 | 2.5 | | | | | |
| Vulcup 40KE | 0.4 | | | | | |
| Tensile Strength | 1528 | 1520 | 1496 | 1555 | 1378 | 1527 |
| 50% Mod | 195 | 221 | 236 | 321 | 307 | 325 |
| 100% Mod | 270 | 636 | 655 | 857 | 750 | 834 |
| 200% Mod | -- | -- | 1485 | -- | -- | -- |
| Elongation % | 197 | 191 | 192 | 171 | 176 | 169 |
| Shore A | 60 | 60 | 60 | 65 | 65 | 65 |
| Specific Gravity | 1.97 | 1.92 | 1.92 | 1.94 | 1.88 | 1.90 |

*A4" indicates polymer aged 4 hrs. @ 300°F.

TABLE 25 BANBURY "BR" MIXES

and COAGENT ADDITION

| R-Number | 211988 | 211992 | 211993 | 211994 |
|-----------------|-----------|-----------|--------|--------|
| | K19730 | K19730 | K19730 | K19730 |
| PNF-200 | | | | |
| Burgess KE | 60 | 60 | 60 | 60 |
| Zeolox 23-A1100 | 10 | 10 | 10 | 10 |
| Elastomag 170 | 8 | 8 | 8 | 8 |
| Stabilizer | 2 | --- | --- | 2 |
| Silane A1100 | --- | 2.5 | 2.5 | --- |
| TAIC | --- | --- | 1.5 | 1.5 |
| Vulcup 40KE | 0.5 | 0.5 | 0.5 | 0.5 |
| f- | 12.22 | 12.22 | 1 | 1 |
| | 4 batches | 5 batches | | |

Cure 30 Min. 150°C
Post Cured 4 hrs. at 175°C (0 indicates no post cure)

| | 0 | X | 0 | X | X | X |
|-------------------------|------|------|------|------|------|------|
| Tensile Str. | 1377 | 1378 | 1475 | 1330 | 1125 | 1414 |
| 50% Mod | 417 | 355 | 424 | 389 | 506 | 393 |
| 100% Mod | 1160 | 1015 | 1223 | 1150 | --- | 1217 |
| Elongation | 120 | 142 | 127 | 118 | 99 | 117 |
| Shore A | 65 | 65 | 65 | 65 | 65 | 65 |
| Tear Strength | | | | | | |
| Die C | | 86 | | 58 | | |
| lbs./ga. (load/gage) | | | | | | |

TABLE 26. WEATHER-O-METER AGING

PNF 200 COMPOUNDS

TEST CONDITIONS: CAM NO. 47 18 HOURS 102 MIN. LIGHT, 18 MIN., WATER
6 HOURS DARK, NO WATER

Unaged Properties:

| | R211 904 | R211 922 |
|------------------|----------|----------|
| Tensile Strength | 1377 | 1330 |
| 50% Modulus | 355 | 369 |
| 100% Modulus | 1015 | 1150 |
| % Elongation | 341 | 118 |
| Shore A | 65 | 65 |

206 Hours (Total Light):

| | | % Change | | % Change |
|------------------|------|----------|------|----------|
| Tensile Strength | 1134 | (-17%) | 1105 | (-16.9%) |
| 50% Modulus | 339 | (-4.5%) | 306 | (-21%) |
| 100% Modulus | 936 | (-7.5%) | 920 | (-19.2%) |
| % Elongation | 125 | (-14.0%) | 123 | (-4.2%) |
| Shore A | 65 | | 65 | |

500 Hours (Total Light):

| | | | | |
|------------------|------|--------|------|----------|
| Tensile Strength | 1168 | (-15%) | 1073 | (-19.3%) |
| 50% Modulus | 387 | (-9%) | 366 | (-5.9%) |
| 100% Modulus | 980 | (-3%) | 987 | (-14%) |
| % Elongation | 127 | (-10%) | 112 | (-5%) |
| Shore A | 65 | 0 | 65 | 0 |

TABLE 27. "FUEL 6" AGING OF MICRO-TENSILE RINGS

R211988
Aging of Rings at 160°F in Fuel "6"

| | Original Properties | Aged 14 Days | % Change | 28 Days | % Change | 42 Days | % Change |
|--------------|------------------------|-----------------|-------------|------------|-------------|------------|-------------|
| Tensile | 1378 | 1092 | (-20.8) | 1054 | (-23.5) | 1043 | (-24.3) |
| 50% Mod | 355 | 177 | (-50.1) | 137 | (+6) | 166 | (53.2) |
| 100% Mod | 1015 | 749 | (-26.2) | 667 | (-34.3) | 684 | (-32.6) |
| Elongation % | 142 | 143.5 | (+1.0) | 145.1 | (+2) | 143 | (+7) |
| Shore A | 65 | 55 | | 55 | | 55 | |

TABLE 28. FUEL CONTAMINATION TESTS

Procedure: Para 4.6.2.1.1 and .2 of MIL T-52573C ASTM D381

| | <u>MIL-T-52573</u> | DESIRED VALUE <u>DAAG46-73-0006</u> | R211988 TEST FUEL <u>Mg/100ml</u> | R21199 TEST FUEL <u>Mg/100ml</u> |
|---|--------------------|---|---|--|
| Unwashed Existent gum Mg/100ml, max. | 60 | 20 | 4.0 | 1.0 |
| Heptane washed existent gum Mg/100ml, max. | -- | 5 | 2.0 | 0.6 |
| Stoved gum, residue Mg/100ml, max. | 20 | -- | 0.6 | -0- |

TABLE 29. FUEL DIFFUSION RATE COATED FABRIC AND VULCANIZATES

Test Fuel: Fuel δ (60v iso octane, 25v toluene, 15v xylenes)

| Stock | Fuel δ Diffusion Rate | ft.-24 hrs. oz./sq. | Coated Fabric Weight | oz./yd. 2 | Vulcanizate Weight | Thickness Mils | Remarks |
|--------------------------|------------------------------------|------------------------|----------------------------|--------------|-----------------------|-------------------|---|
| 211988 | 0.21 | ft.-24 hrs. oz./sq. | --- | oz./yd. 2 | --- | 29. | } wrap cured in oven 30' 3500F, 4 hrs. 3500 TAIC Coagent in 211988 TAIC Coagent in 211992 |
| 211988 | 0.09 | --- | --- | --- | 42.4 | 60 | |
| 211994 | 0.12 | --- | --- | --- | 93.6 | 51 | |
| 211993 | 0.10 | --- | --- | --- | 71.0 | 51 | |
| 211992 | 0.095 | --- | --- | --- | 71.6 | 55 | 1508-37 wrap cured fabric treatment: |
| 211992 | 0.036 | 89.2 | --- | --- | 83.1 | 66 | { TX-9 Thixon 300/301 TX-10 Thixon 300/301 on PNF-200 dispersion purified polymer: TX-11 Thixon 300/301 TX-12 Thixon 300/301 |
| 211992 | 0.023 | 92.0 | --- | --- | --- | 68 | |
| 211974 | 0.01 | 78.5 | --- | --- | --- | 59 | |
| 211975 | 0.01 | 70.0 | --- | --- | --- | 52 | |
| 211992 | 0.041 | 93.1 | --- | --- | --- | 72 | TX-13 3.4% T.S. Thixon 300/301 Nylon Backing |
| 211992 | 0.042 | 94.8 | --- | --- | --- | 67 | TX-14 3.4% T.S. Thixon 300/301 |
| 211992 | 0.05 | 73.8 | --- | --- | --- | 58 | TX-15A 3.4% T.S. Thixon 300/301 |
| WO-05504 Type II Fuel | 0.096 | 33.9 | --- | --- | --- | 47 | Coated Fabric Reported to meet Mil T-52573C Fuel Diffusion Requirements |
| | 0.107 | 33.7 | --- | --- | --- | 47 | |
| WO-03305 Type II | 0.037 | 34.1 | --- | --- | --- | 44 | |
| | 0.050 | 34.1 | --- | --- | --- | 44 | |

TABLE 30. FUEL DIFFUSION TESTS OF COATED FABRICS WITH VARIOUS FABRIC SURFACE TREATMENTS

| <u>Stock</u> | <u>Fuel & Diffusion Rate</u> | <u>Coated Fabric Weight</u> | <u>Thickness Mils</u> | <u>Remarks</u> |
|--------------|--|-------------------------------------|---------------------------|----------------|
| | fl./oz/sq. ft.- 24 hrs. | oz/yd ² | | |
| 211992 | 0.052 | 60.9 | 45 | Thixon A/B |
| 211992 | 0.10 | 58.0 | 45 | Chemlok AP134 |
| 211992 | 0.10 | 59.6 | 45 | Chemlok 607 |
| 211992 | 0.096 | 60.6 | 45 | Fluorel FC5150 |

TABLE 21. BREAKING STRENGTH OF COATED FABRICS WITH VARIOUS FABRIC SURFACE TREATMENTS

Stock: 211992

| | | | Aged at 160°F | | Aged at 160°F | | Fabric Surface Treatment |
|---------|------------|-----------|----------------|-----------|------------------|-----------|--------------------------|
| Initial | lbs/in | thickness | 8 Days - Water | | 8 Days in Fuel δ | | |
| | | | lbs/in | thickness | lbs/in | thickness | |
| | | | initial | final | initial | final | |
| 1B | 311 | 43 mils | 300 | 47 | 239 | 45 | Thixon 300/30 |
| | 350 | | 300 | 56 | 279 | 46 | |
| Average | <u>334</u> | | <u>300</u> | | <u>239</u> | (-28%) | |
| 2B | 175 | 44 | 214 | 46 | 160 | 44 | Chemlok AP 134 |
| | 171 | | 212 | 46 | 161 | 44 | |
| Average | <u>180</u> | | <u>213</u> | | <u>160</u> | (-8%) | |
| 3B | 282 | 44 | 310 | 46 | 217 | 42 | Chemlok 607 |
| | 282 | | 285 | 46 | 185 | 44 | |
| Average | <u>255</u> | | <u>298</u> | | <u>201</u> | (-26%) | |
| 4B | 371 | 44 | 275 | 45 | 286 | 43 | Fluorel PC5150 |
| | 373 | | 283 | 45 | 261 | 45 | |
| Average | <u>380</u> | | <u>279</u> | | <u>273</u> | (-27%) | |

TAB. 32. REPEAT MIXES AND MIXER FILLING FACTOR CHECK

| R-Number 211, | 995 (944) | 996 (945) | 997 (946) | 998 | 999 |
|----------------------------|-----------------|-----------------|-----------------|--------|--------|
| PNE-200 | 19736 | K19861 | K19862 | K19736 | K19736 |
| Tullanox 500 | 15 | | | | |
| Zeolox 23 A1100 | 5 | | | | |
| Elastomag 170 | 6 | | | | |
| Stabilizer | 2 | | | | |
| Silane A174 | 2.5 | | | | |
| Vulcup 40KE | 0.4 | | | | |
| filling factor | 0.6 | 0.6 | 0.6 | 1 | 0.8 |
| f= | 20,000 M-gm-min | | | | |
| Σ Torque | | | | | |
| Max Temp. | 85°C | 81°C | 75°C | 97°C | 90°C |
| Cure 30 min at 150°C | | | | | |
| Post cure 4 hours at 178°C | | | | | |
| Tensile Strength | 1439 | 1711 | 1985 | 1957 | 1911 |
| 50% Modulus | 164 | 107 | 141 | 183 | 174 |
| 100% Modulus | 384 | 294 | 394 | 533 | 461 |
| 200% Modulus | 1114 | 1094 | 1410 | 1640 | 1392 |
| Elongation % | 233 | 251 | 246 | 226 | 246 |
| Shore A | 55 | 50 | 55 | 55 | 55 |
| Specific Gravity | 1.9 | 1.9 | 1.9 | 1.9 | 1.9 |
| Tensile Strength | R211944 1797 | R211945 1742 | R211946 1813 | | |
| 50% Modulus | 221 | 144 | 148 | | |
| 100% Modulus | 644 | 346 | 375 | | |
| 200% Modulus | -- | 1128 | 1109 | | |
| Elongation % | 189 | 257 | 272 | | |
| Shore A | 55 | 55 | 55 | | |
| f=1 | | | | | |

TABLE 33. ADHESIVE PEEL STRENGTH TESTS OF PNF[®]-200 COATED FABRIC

2x2 NYLON BASKET WEAVE 8.5 oz./yd²

| Stock | Fabric Surface Treatment | Coated Fabric Thickness | 180° Peel Strength |
|--------|--|----------------------------|--|
| 211988 | PNF-200 211992 dispersion +0.25 Vulcup 40KE + 0.25 TAIC | 68 69 72 72 | A-1 1.0 ppi A-2 1.05 A-3 1.05 A-4 1.1 |
| 211992 | AS ABOVE | 70 74 69 72 68 | A-5 2.04 A-6 2.33 A-7 2.00 A-8 1.9 A-9 1.48 |
| 211988 | Thixon A/B | 68 | TS-88A 8.7 TS-88B 7.6 |
| 211988 | Nylon 8061 + 211992 dispersion Nylon 8064 + 211992 dispersion | 68 68 70 70 | N-1A 1.0 N-1B 1.0 N-2A 1.0 N-2B 0.9 |
| 211992 | Resorcinol-formaldehyde - Vinyl pyridine latex FRS-262 Ammonium hydroxide | 77 | AR-1A 1.0 AR-1B 0.9 |
| 211988 | Resorcinol-formaldehyde vinyl pyridine latex FRS-262 Sodium hydroxide C-103 Fabric | 55 | R-1A 3.0 R-1B 2.5 |
| 211988 | Thixon A/B 1:10 dilution Thixon A/B 1:100 dilution Chemlok 607/Z6020 | 71 66 65 | TX-3A 1.5 TX-3B 1.8 TX-4A 0.9 TX-4B 0.9 CH-1A 1.1 CH-1B 1.0 |

TABLE 34. ADHESIVE PEEL STRENGTH TESTS OF PNF[®]-200 COATED FABRIC

| 2x2 NYLON BASKET WEAVE | | Coated Fabric Thickness | 180° | |
|------------------------|--|----------------------------|---------------|------|
| Stock | Fabric Surface Treatment | | Peel Strength | |
| 211988 | Thixon A/B No post cure | 68 | 988-S-A | 7.3 |
| | | | 988-S-B | 6.3 |
| | | | TS-88A | 9.5 |
| | | | TS-88B | 8.8 |
| 211992 | Thixon A/B | 68 | TS-92A | 4.4+ |
| | | | TS-92B | 4.2 |
| 211996 | Thixon A/E 1:10 dilution | 60 | TX-1A | 2.1 |
| | | | TX-1B | 2.1 |
| | Thixon A/B 1:10 dilution | 55 | TX-2A | 4.7 |
| | | | TX-2B | 5.1 |
| 211992 | Chemlok AP133 | 65 | CH133-1 | 3.2 |
| | Chemlok AP133 1:1 methanol dilution | | CH133-2 | 3.6 |
| | Chemlok AP133 2 hr. air dry | | | 2.3 |
| | Thixon A/B + PNF-200 165°C 30 min. Nylon G103 backing 4 hr. post cure 350°F | 67 | TX-9 | 7.8 |
| | | 68 | TX-10 | 5.5 |

TABLE 15. ADHESIVE PEEL STRENGTH AND BREAKING STRENGTH
OF COATED FABRIC

| Stock | Thickness Mils | Breaking Strength | 180° Peel Strength | Remarks | Fabric Surface Treatment |
|--------|-------------------|----------------------|--------------------------|---------|-----------------------------------|
| 211992 | 67 | 370 lbs/in | 6.3 ppi | TX5A | Thixon 30 min 150°C press cure |
| | 66 | 350 | 5.95 | | |
| | 72 | 420 | 4.5 | TX6A | Thixon 30 min 180°C press cure |
| | 71 | 407 | 4.5 | TX6B | |
| | 70 | 480 | 5.7 | TX7A | Thixon 30 min 170°C press cure |
| | 70 | 375 | 5.95 | TX7B | |
| | 65 | 280 | 5.5 | TX8A | Thixon 30 min 150°C press cure |
| | 66 | 280 | 5.5 | TX8B | +Zonyl FSN |

TABLE 55. STABILIZER MASTERBATCH FORMULATION

| | | 219600 | 219606 |
|----------------------|--------|--------|--------|
| DSE-200 | K19736 | 67.2 | -- |
| | K19862 | 32.8 | 100 |
| Burgess KE | | 20 | 20 |
| Zn II bis(quinolate) | | 40 | 40 |
| | | 3.29 | 3.29 |
| | | 1 | 2 |

Burgess KE - ZnII bis(quinolate) ball milled to reduce particle size of stabilizer.

TABLE 57. BANBURY "BR" MIXES OF PNF[®]-200

| R-Number | 219601 | 219602 | 219607 | 219608 | 219609 |
|---|--------|--------|--------|--------|---------|
| PNF-200 | K19736 | K19862 | K19861 | K19861 | 1508-49 |
| Burgess KE | 55 | 55 | -- | 60 | 60 |
| Tullanox 500 | -- | -- | 15 | -- | -- |
| Zeolox 23-A1100 | 8 | 8 | 5 | 10 | 10 |
| Elastomag 170 | 8 | 8 | 6 | 8 | 8 |
| Silane A1100 | 1 | 1 | 1.5 | 1.5 | 1.5 |
| Stabilizer as | | | | | |
| R219600 | 8 | 8 | -- | -- | -- |
| R219606 | -- | -- | 4 | 4 | 4 |
| Vulcup 40KE | 0.4 | 0.4 | 0.4 | 0.5 | 0.5 |
| No. of Batches | 3 | 2 | 4 | 5 | 3 |
| loading factor f= | 12.59 | 12.59 | 16.68 | 11.96 | 11.96 |
| Max Temp. during mix | 172°F | 172°F | 152°F | 165°F | 165°F |
| Batches Combined Labeled 219601 | | | | | |
| Monsanto Rheometer Data T=340°F, 1° Arc 100 RPM, Mini die | | | | | |
| Scorch Time | 4.2 | | 4.7 | 3.7 | 3.5 |
| Opt cure TC(90) | 17.5 | | 14.0 | 20.0 | 20.0 |
| Min Torque | 9.9 | | 6.5 | 10.3 | 10.1 |
| Torque at 90% cure | 15.7 | | 10.5 | 17.0 | 17.2 |
| Max Torque | 16.3 | | 10.9 | 17.7 | 18.0 |
| Cure Rate Index | 7.5 | | 10.8 | 6.1 | 6.1 |
| Cure 30 min at 340°F 4 hrs at 350°F | | | | | |
| Ring Tensile Data | | | | | |
| Tensile Strength | 1392 | | 1562 | 1303 | 1309 |
| 50% Modulus | 241 | | 71 | 286 | 292 |
| 100% Modulus | 849 | | 211 | 872 | 917 |
| 200% Modulus | -- | | 797 | -- | -- |
| Elongation | 157 | | 294 | 158 | 140 |
| Shore A | 60 | | 45 | 65 | 65 |
| Specific Gravity | 2.1 | | 1.9 | 2.1 | 2.1 |

TABLE 28. COMPARISON OF PHYSICAL PROPERTIES OF VULCANIZATES WITH PURIFIED POLYMER 1508-28

| | R211974 | R211975 | R219603 | R211924 |
|-----------------------------|---------|---------|---------|----------|
| PNF-200 | 1508-28 | 1508-28 | 1508-28 | K19736 |
| Burgess KE | 60 | 60 | 60 | 60 |
| Zeolox 23-A1100 | 10 | 10 | 10 | 10 |
| Elastomag 170 | 8 | 8 | 8 | 8 |
| Stabilizer R211855 | 2 | -- | -- | 2 |
| Vulcup 40KE | 0.5 | 0.5 | 0.5 | .5 |
| Cure 30' 150°C 4 hrs. 176°C | | | | |
| Tensile Strength | 1482 | 1459 | 1185 | 1464 |
| 50% Mod | 464 | 425 | 354 | 467 |
| 100% Mod | 1371 | 1316 | 1141 | 1362 |
| 200% Mod | -- | -- | -- | -- |
| Elongation % | 109 | 112 | 107 | 110 |
| Shore A | 65 | 65 | | 65 |
| Specific Gravity | | 2.1 | | |
| Diffusion Rate Fuel | 0.1 | 0.1 | 0.052 | 0.087* |
| Coated Fabric Weight | 78.5 | 70 | 60 | 78* 56.5 |

A 14 wt.% PNF-200, K198Cl, polymer solution (methanol), filtered, coagulated in water, and vacuum dried (1508-28).

| | <u>K19861</u> | <u>1508-28</u> |
|------------|---------------|----------------|
| DSV (dl/g) | 2.3 | 2.3 |
| gel (%) | 0.0 | 0.0 |
| Na (wt.%) | 0.014 | 0.0 |
| Cl (wt.%) | 0.022 | 0.0 |

*Vulcanizate wt. oz/yd²

TABLE 39. COMPARISON OF PHYSICAL PROPERTIES OF K19861
VS PURIFIED POLYMER (1508-49)

| Stock | 219608 | 219609 |
|-------------------------------|---------------|---------------|
| Original: | | |
| Tensile Strength | 1303 | 1309 |
| 50% Modulus | 286 | 292 |
| 100% Modulus | 872 | 917 |
| Elongation at Break | 158 | 140 |
| Shore A | 65 | 65 |
| Aged: Fuel 8 3 Days at 160°F | | |
| Tensile Strength | 1139 (-12.5%) | 1145 (-12.5%) |
| 50% Modulus | 194 (-32%) | 159 (-45.5%) |
| 100% Modulus | 773 (-11.4%) | 765 (-16.6%) |
| Elongation at Break | 143 (-9.5%) | 136 (-2.8%) |
| Shore A | 60 (-7.7%) | 60 (-7.7%) |
| Aged: Fuel 8 14 Days at 160°F | | |
| Tensile Strength | 1112 (-14.6%) | 1192 (-8.9%) |
| 50% Modulus | 210 (-26.5%) | 232 (-20.5%) |
| 100% Modulus | 807 (-7.5%) | 884 (-3.5%) |
| Elongation at Break | 135 (-14.6%) | 132 (-2.8%) |
| Shore A | 60 (-7.7%) | 60 (-7.7%) |

FUEL DIFFUSION TESTS - VULCANIZATES AND COATED FABRICS

TABLE 40.

| Stock | Fuel δ Diffusion Rate | Coated Fabric | Vulcanizate Weight | Thickness | Fabric Surface Treatment |
|--------|------------------------------------|------------------|-----------------------|-----------|--|
| | Fl. oz/sq ft-24 hrs | oz/sq. yd | oz/sq. yd | Mils | |
| 219501 | 0.09 | -- | 90 | 50 | A-Thixon 300/301 3.8% F. B-Fluorel 5150 |
| 219601 | 0.07 | 54 | -- | 48 | |
| 219601 | 0.12 | 57 | -- | 45 | |
| 219607 | 0.19 | -- | 87 | 64 | A B |
| 219607 | 0.08 | 55 | -- | 45 | |
| 219607 | 0.18 | 53 | -- | 45 | |
| 219608 | 0.08 | -- | 87 | 58 | A B |
| 219608 | 0.06 | 58 | -- | 45 | |
| 219608 | 0.09 | 62 | -- | 47 | |
| 219509 | 0.08 | -- | 87 | 58 | A B |
| 219609 | 0.06 | 63 | -- | 48 | |
| 219609 | 0.10 | 60 | -- | 46 | |

TABLE 7-1. BREAKING STRENGTH OF WATER AGED COATED FABRICS

| Stock | Surface Treatment | Thickness ozs/sq. yd Mils | | 7 Days at 160°F in DI water | |
|--------|-------------------|------------------------------|----|-----------------------------|-----|
| | | | | Breaking Strength | psi |
| 219601 | Thixon 300/301 | 48 | 64 | | 240 |
| | Fluorel 5150 | 45 | 57 | | 337 |
| 219607 | Thixon | 45 | 55 | | 370 |
| | Fluorel | 45 | 53 | | 383 |
| 219608 | Thixon | 45 | 58 | | 288 |
| | Fluorel | 47 | 62 | | 327 |
| 219609 | Thixon | 48 | 63 | | 345 |
| | Fluorel | 46 | 60 | | 340 |

TABLE 42. PHYSICAL PROPERTIES OF - WRAP CURED CALENDERED SHEET

Stock: R211992

Calendered Thickness: 0.055-0.053"

Calendered Width: 10"

Cured: 30 min. at 350°F

Wrapped around aluminum tube - interleaved .005" steel shim.

Check of stress-strain properties along strip every six inches.

Post Cured: Un-wrapped 4 hours 350°F oven

| | <u>1508-37-1</u> | <u>1508-37-2</u> | <u>1508-37-3</u> | <u>1508-37-4</u> |
|--------------------------|------------------|------------------|------------------|------------------|
| Tensile Str. | 1212 | 1257 | 1275 | 1298 |
| 50% Modulus | 389 | 379 | 404 | 400 |
| 100% Modulus | 1132 | 1127 | 1169 | 1196 |
| % Elongation at Break | 109 | 114 | 115 | 110 |
| Shore A | 60 | 60 | 60 | 60 |
| Specific Gravity | 2.1 | 2.1 | 2.1 | 2.2 |

Press Cured 30 min. at 350°F 4 hours at 350°F

| | <u>R211992</u> |
|--------------|----------------|
| Tensile Str. | 1130 |
| 50% Modulus | 389 |
| 100% Modulus | 1150 |
| Elongation | 118 |
| Shore A | 65 |

TABLE 43. SEAM ADHESION PEEL STRENGTH

Press Cured Adhesion Pads

| Thixon Treated Fabric | Initial Strength | Fabric - To Rubber Strength |
|-----------------------|------------------|-----------------------------|
| 211988 - 1 | 8.2 ppi | |
| 2 | 9.0 | |
| 3 | 8.5 | |
| 4 | 8.7 | |
| 5 | 8.6 | |
| 6 | 7.8 | |
| 7 | 8.2 | |

Thixon Treated Fabric:
Press Cured Adhesion Pads

| | | |
|--------------|----|---------|
| 211988 - 2-1 | -- | 2.7 ppi |
| 2-2 | -- | 6.0 |

Thixon/Fluorel 5150 Treated Fabric:

| | | | |
|------------|---------|-----|--------------|
| 219601 - 1 | 7.7 ppi | 6.0 | seam failure |
| 2 | 8.8 | 6.2 | " " |
| 3 | 9.3 | 6.2 | " " |

Wrap Cured Seam - Fluorel Treated

| Thixon/Fluorel Treated Fabric | |
|-------------------------------|---------|
| 219601 - 1 | 9.6 ppi |
| 2 | 9.2 |
| 3 | 8.8 |
| 4 | 9.2 |

Press Cured Seam

Calender Fabric Treated with 5% 219607 in MIBK

Seam Treatment: Chemlok 607 Spray

| | | |
|------------|------|---------|
| 219608 - 1 | 10.8 | 6.0 ppi |
| 2 | 10.4 | 6.0 |
| 3 | 8.0 | 5.0 |

Seam Treatment: Acetone Wipe

| | | |
|------------|--------|-----|
| 219608 - 1 | 12 ppi | 3.0 |
| 2 | 12 | 5.0 |
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Army Materials and Mechanics Research Center
Watertown, Massachusetts 02172
HYPERBOLICALLY FLAME-RETARDANT COATED FABRIC
FOR COLLAPSIBLE PULP STORAGE TANKS
Richard V. Allen and George E. Mitchell
Firestone Central Research Laboratories
1200 Firestone Parkway, Akron, OH 44317
Final Report MMRC 77-14, July 1977, 65 pp illus -
tables; Contract MACD-76-2-0026; S/A Project:
1545102071; ARCS Code: 67402; Agency
Accession: DA06788

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DECLASSIFICATION

Key Words
Thermoplastic polymers
Polyphosphazenes
Phosphonitrilic flame-retardant
Coated fabrics
Fuel tanks
Storage tanks

This report describes the preparation of fuel resistant
coated fabric materials that are serviceable at
temperatures as low as -70°F. The coated fabric materials were made from a phosphonitrilic
flame-retardant compound coated on nylon fabric. Efforts were directed toward attainment
of considerable compound and test evaluations of the coated fabric. High filler loaded
materials proved to be better previously materials. These showed good diffusion rates with
test fluids (20-4-75, Type II and Fuel 5). Even better diffusion rates were seen with
Aeris diethyl fuel. The low temperature flexibility of the voluminates and coated fabric
was promising for 100-300 compounds. The same adhesion strength and the fabric-to-rubber
adhesion need to be improved before utilization of this material in tank construction.

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